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## **VOLUME I FINAL REPORT**

# **EFFECT OF ENVIRONMENT ON INSULATION MATERIALS**

**By**

**R.T. Parmley, F.J. Smith, A.P. Glassford, J. Coleman and D.R. Stevenson**

**LOCKHEED MISSILES & SPACE COMPANY, INC.**  
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16. Abstract Twenty candidate multilayer insulation and insulation related materials were subjected to eight conditions that represent possible operational environments. These exposures include ground contaminants, various operational temperatures, space vacuum, space-vented propellants, and tank leakage. The objective of this program was to obtain and evaluate the data from these exposures to provide both a quantitative and qualitative description of the degradation to certain physical and thermal properties, and from this, to obtain a better understanding of the environmental effects on the insulation performance. Extensive tabular and graphical test results are provided on the effects of the environmental exposures on material properties along with listings of insulation materials recommended for use in: <ul style="list-style-type: none"> <li>o Oxygen and fluorine tank insulation systems</li> <li>o Vacuum, purged and unprotected insulation systems</li> <li>o Specific environments and exposure times</li> </ul>					
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## FOREWORD

The Lockheed Missiles & Space Company, Inc. (LMSC) is submitting this final report in partial fulfillment of the requirements of Contract NAS 3-14342, Effect of Environments on Insulation Materials. Mr. R. T. Parmley was the LMSC Program Manager and Mr. James R. Barber was the NASA-Lewis Research Center Project Manager. The work is presented in two volumes. The first volume (NASA CR 120978) covers the total scope of the program, a summary and discussion of the results plus significant conclusions developed from the results. Volume II (NASA CR 120979) is basically a Materials Property Handbook providing the detailed test results in tabular and graphical form.

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## Section 0

### SUMMARY

Twenty different multilayer insulation or insulation related materials were selected for test on this program. The materials include aluminized and goldized Mylar and Kapton radiation shields, low conductivity spacers of silk net, Nylon net, Dacron net and Tissuglas, a glass cloth, two adhesives, polyurethane foam, Velcro fasteners, Teflon film and two thermal control paints.

The properties include weight, density, coating adhesion, flexibility, tensile strength, shear strength, peel strength, compression strength, layer density, emittance, solar reflectance, solar absorbtance, fluorine and oxygen compatibility and outgassing rates.

The test data were reduced and then tabulated and plotted in a Materials Property Handbook as Volume II (NASA CR 120979) containing approximately 1500 tables and graphs. For each property value measured after environmental exposures, non-exposed control tests were also performed to provide a reference against which the environmental effects could be judged. Where significant effects did occur, percentage changes were calculated and analyzed.

Recommendations were then made on which materials were suitable for use in insulation systems (1) on oxygen or fluorine tanks, (2) in vacuum, purged and unprotected insulation systems and (3) in different environments and for specific exposure times.

The magnitude of the test matrix does not permit a complete summation here of all the major conclusions reached during the test program. However, some of the more important findings of the program are given below.



- ° Double coated aluminum and gold Mylar films are fluorine compatible for short durations. Gold coatings provide greater protection to the substrate than aluminum.
- ° Double coated gold Mylar films are compatible for short periods with moist air/fluorine environments.
- ° Kapton is more resistant to fluorine attack than Mylar.
- ° Gold coated films are recommended for use in salt air and high humidity environments.
- ° Kapton outgasses less than Mylar.
- ° Gold outgasses less than aluminum.
- ° Dacron net exhibited the fewest property changes of the four spacers tested.
- ° Silk net (with fire retardent sizing) is compatible with dry fluorine environments.
- ° The Narmco 7343/7139 polyurethane adhesive exhibited the fewest property changes among the adhesives tested. The Goodyear 4001/4004 adhesive is compatible with dry fluorine for short time periods.
- ° Polyester Velcro fasteners and Teflon film properties are stable in the various test environments; Teflon is fluorine compatible; Velcro fasteners are attacked.
- ° Properties of the black thermal control paint are extremely stable in all environments (except fluorine).

- The white Thermatrol paint exhibited some small property changes after some of the exposures. (It is also attacked by fluorine.)
- The majority of the property changes measured occurred in the high temperature 200°F (93°C) environments at one atmosphere or in vacuum, the salt air environment and the different fluorine environments.

## Section 1

### INTRODUCTION

Cryogenic liquids already play a significant role in the nation's space program as propellants in the various Saturn stages. As the emphasis is shifted to longer mission durations (requiring high performance insulations) and reusable vehicle systems such as the Space Shuttle, with life cycles of up to 100 flights and a use span of up to ten years, the long term structural and thermal performance stability of the insulation systems becomes of key importance. Since the shuttle system must operate both within and outside the atmosphere, the insulations can experience a wide variety of environments during manufacture, prelaunch, ascent, orbit, reentry and storage. It is important to know the effects these environments have on the insulation materials so (1) the more stable materials can be selected that do not require replacement, (2) the performance band can be established for its operating life time, and (3) the requirements for maintaining inert atmospheres between or during portions of the flight are minimized.

For example, limited prior tests (Ref. 1) have indicated moist, condensing environments remove aluminized coatings from multilayer insulation radiation shields but not gold coated shields. If the aluminized system was exposed to this type of environment between flights, i.e., moisture condensation at night, the emittance may increase drastically and the thermal performance of the insulation is degraded. The added cost of maintaining an inert atmosphere in the insulation between flights must be added to the cost of this system. On the other hand, if gold coated mylar is substituted, the radiation shield material costs increase by a factor of seventy. Consequently, quantitative test data are required on the effects of environmental exposure so the more cost effective material can be selected for a particular application.

Twenty different multilayer insulation or insulation related materials were selected for test on this program. The test matrix performed for each material is summarized in the following four categories: multilayer radiation shields (Table 1), multilayer spacer materials (Table 2), ground-hold insulation materials (Table 3), and miscellaneous insulation materials (Table 4).

The low emittance radiation shield materials are used to reduce radiation heat transfer through the insulation; the low conductivity spacers interspersed between the radiation shields decrease the solid conduction component of heat transfer. In the ground-hold insulation material class, the beta glass cloth has been proposed for use in meteoroid shields and purge bag reinforcements. The two adhesives may be used to bond insulation attachments to the tank and the polyurethane foam is a candidate for a sublayer insulation (beneath the multilayers). In the miscellaneous insulation material class, the velcro fasteners are used to attach insulation blankets to the tank (as well as one blanket to another), the teflon film is used in purge bags and in buttons, threads and tabs used for tying blankets together. The thermal control paints are used for passive low (Thermatrol) or high (Series 400 Black Paint) temperature control in orbit.

Each of these materials are exposed to 8 different conditions (including a control environment for reference) representing operational environments that include different combinations of high humidity, salt air, water immersion, various operational temperatures, space vacuum, space-vented propellants and tank leakage. The effect of these environments on the specified properties shown in Tables 1 through 4 are determined as a function of exposure time, exposure temperature or in some cases fluorine or oxygen partial pressure.

The report is organized in six sections. Identification of the test materials and test specimen designs is given first followed by a description of each environmental exposure condition; the pre-exposure, in-situ and post exposure test procedures are described next; finally, the test results, discussion of results and conclusions are provided.





TABLE 1 - TEST MATRIX FOR MULTILAYER INSULATION RADIATION SHIELDS (continued 3)

Propellants***	Exposure Environment			Materials (All 1/4 MIL)	Pre-exposure Tests (2 Ea for Each Material Specimen Weighed Before and After Exposure; Total No. of Data Points Shown in Parentheses)	In-Situ Tests	Post-exposure Tests (2 Ea for Each Material Specimen Weighed Before and After Exposure; Total No. of Data Points Shown in Parentheses)	No. of Test Specimens
	Back-Fill Conditions							
	Gas/Temperature °F/°C	Pressure torr (n/m <sup>2</sup> )	Back-Fill Conditions Partial Pressure torr (n/m <sup>2</sup> )					
a.	-70(21)	10 <sup>-6</sup> (1.33 x 10 <sup>-1</sup> )	CO <sub>2</sub>	760 (1.02 x 10 <sup>5</sup> )	VS(1)	VS(1)	VS(1)	2
b.	"	"	GF <sub>2</sub>	"	"	"	"	2
c.	-70(21)	10 <sup>-6</sup> (1.33 x 10 <sup>-1</sup> )	CO <sub>2</sub>	10 <sup>-3</sup> (0.133)	"	"	W(1), F <sub>1</sub> (2), F <sub>1</sub> (2), Al(2), c(2), q(2)	4
d.	"	"	"	"	"	"	W(1), F <sub>1</sub> (2), F <sub>1</sub> (2), AD(2), c(2), q(2)	4
e.	"	"	GF <sub>2</sub>	"	"	"	"	4
f.	"	"	"	"	"	"	"	4
g.	Air, 95 (35) 95% R. H.	760 (1.02 x 10 <sup>5</sup> )	GF <sub>2</sub>	10 <sup>-3</sup> (0.133)	"	"	"	24
h.	"	"	"	760 (1.02 x 10 <sup>5</sup> )	"	"	"	24
TOTAL								548

Legend

Legend

- AD Coating Adhesion
- F<sub>0</sub> Flexibility
- F<sub>1</sub> Tensile Strength
- IC Ignition (Chamber Pressure Rise)
- IC Ignition (Chamber Pressure Rise)
- VS Visual Observation
- W Weight
- W Solar Transmittance of Uncoated Side
- W Total Normal Emissance of all Coated Sides

\*\*Each outgassing measurement and product identification counted as one data point; one specimen per material tested.  
\*\*\*Only those materials which do not ignite are continued in test sequence shown.

TABLE 2 - TEST MATRIX FOR MULTILAYER INSULATION SPACER MATERIALS

Exposure Environment	Materials SN - Silk Net NN - Nylon Net DN - Dacron Net TG - Tiaraglas	Pre-Exposure Tests (2 Ea for Each Material Specimen Required Before and After Exposure - Total No. of Data Points Shown in Parentheses)	In-situ Tests	Post-Exposure Tests (2 Ea for Each Material Specimen Required Before and After Exposure - Total No. of Data Points Shown in Parentheses)	No. of Test Specimens
1. Control, Held at 70°F (21°C) Exposure Time (Days) For Each Lot Received { 0 (as received) 10 150	SN, NN, DN, TG	W(4), F <sub>c</sub> (4), F <sub>t</sub> (8) "			12 12 12
2. Vacuum Environment $\leq 10^{-6}$ torr ( $\leq 1.33 \times 10^{-4}$ n/m <sup>2</sup> ) Temperature Exposure Time O <sub>2</sub> N <sub>2</sub> 660 386 24 660 386 240 (170 for OG) 530 294 240 (170 for OG) 140 78 240 (170 for OG) 37 21 240		W(8) " " " "	OG(48)** OG(48)** OG(48)**	W(4), F <sub>c</sub> (4), F <sub>t</sub> (8) " " " "	12 16 16 16 12
3. High Temperature, 200°F (93°C), 40% R.H. Exposure Time (hr) 24 24 240		W(4) " "		W(4), F <sub>c</sub> (4), F <sub>t</sub> (8) " "	12 12 12
4. Vacuum, Gas Purge, Vacuum Vacuum $\leq 10^{-6}$ torr ( $\leq 1.33 \times 10^{-4}$ n/m <sup>2</sup> ) 200°F (93°C) Exposure Time (hr) 6 6 170 170 Gas Exposure Time (hr) CH <sub>2</sub> 4 CH <sub>4</sub> 4		W(4) " "	OG(48)**	W(4), F <sub>c</sub> (4), F <sub>t</sub> (8) " "	4 4
5. 95% Humidity at 95°F (35°C) Exposure Time (hr) 12 24 72		W(4) " "		W(4), F <sub>c</sub> (4), F <sub>t</sub> (8) " "	12 12 12

Legend  
F<sub>c</sub> Layer Compression Tests  
F<sub>t</sub> Tensile Strength  
F<sub>1</sub> Ignition (Chamber Pressure Rise)  
OG Gas Purge and Products (once each hr, first 8 hr, once every 21 hr thereafter)  
VB Visual Observation  
W Weight

\*\*Each outgassing measurement and product identification counted as one data point; one specimen per material tested.  
\*\*\*Or until ignition occurs  
\*\*\*\*Only those materials which do not ignite are continued in test sequence shown.



TABLE 2 - TEST MATRIX FOR MULTILAYER INSULATION SPACER MATERIALS (continued 2)

Exposure Environment	Materials SN - Silk Net NN - Nylon Net TG - Tiesaglas	Pre-Exposure Tests (2 Ea for Each Material: Specimens Weighed Before and After Exposure - Total No. of Data Points Shown in Parentheses)	In-situ Tests	Post-Exposure Tests (2 Ea for Each Material: Specimens Weighed Before and After Exposure - Total No. of Data Points Shown in Parentheses)	No. of Test Specimens																																																																		
6. 95% Humidity at 95°F (35°C), Salt Air Exposure Time (hr) 12 24 72	SN, NN, UN, TG	W(4) "		W(1), F <sub>c</sub> (4), F <sub>i</sub> (4) "	12 12 12																																																																		
7. Water at Room Temperature Exposure Time (hr) 0.5 4 24		W(4) "		W(4), F <sub>c</sub> (4), F <sub>i</sub> (4) "	12 12 12																																																																		
8. Propellants***																																																																							
<table border="1"> <thead> <tr> <th colspan="2">Initial Chamber Conditions</th><th colspan="2">Back-Fill Conditions</th><th colspan="2">Back-Fill</th></tr> <tr> <th>Gas/Temperature</th><th>Pressure</th><th>Gas</th><th>Partial Pressure</th><th>Exposure Time, Maximum**</th><th></th></tr> <tr> <th>P<sub>f</sub> (°C)</th><th>torr (n/m<sup>2</sup>)</th><th></th><th>torr (n/m<sup>2</sup>)</th><th></th><th></th></tr> </thead> <tbody> <tr> <td>a. -70 (21)</td><td>10<sup>-6</sup> (1.33 x 10<sup>-4</sup>)</td><td>CO<sub>2</sub></td><td>760 (1.02 x 10<sup>5</sup>)</td><td>To Ignition (or 1 ATM pressure)</td><td></td></tr> <tr> <td>b. -70 (21)</td><td>10<sup>-6</sup> (1.33 x 10<sup>-4</sup>)</td><td>GF<sub>2</sub></td><td>10<sup>-3</sup> (0.133)</td><td>100 hr</td><td></td></tr> <tr> <td>c. -70 (21)</td><td>"</td><td>CO<sub>2</sub></td><td>"</td><td>150 days</td><td></td></tr> <tr> <td>d. -70 (21)</td><td>"</td><td>GF<sub>2</sub></td><td>"</td><td>100 hr</td><td></td></tr> <tr> <td>e. -70 (21)</td><td>"</td><td>GF<sub>2</sub></td><td>"</td><td>150 days</td><td></td></tr> <tr> <td>f. -70 (21)</td><td>"</td><td>GF<sub>2</sub></td><td>"</td><td>150 days</td><td></td></tr> <tr> <td>g. AIF, 95 (35)</td><td>760 (1.02 x 10<sup>5</sup>)</td><td>GF<sub>2</sub></td><td>10<sup>-3</sup> (0.133)</td><td>4 hr</td><td></td></tr> <tr> <td>h. 95% R. H.</td><td>"</td><td>"</td><td>760 (1.02 x 10<sup>5</sup>)</td><td>"</td><td></td></tr> </tbody> </table>						Initial Chamber Conditions		Back-Fill Conditions		Back-Fill		Gas/Temperature	Pressure	Gas	Partial Pressure	Exposure Time, Maximum**		P <sub>f</sub> (°C)	torr (n/m <sup>2</sup> )		torr (n/m <sup>2</sup> )			a. -70 (21)	10 <sup>-6</sup> (1.33 x 10 <sup>-4</sup> )	CO <sub>2</sub>	760 (1.02 x 10 <sup>5</sup> )	To Ignition (or 1 ATM pressure)		b. -70 (21)	10 <sup>-6</sup> (1.33 x 10 <sup>-4</sup> )	GF <sub>2</sub>	10 <sup>-3</sup> (0.133)	100 hr		c. -70 (21)	"	CO <sub>2</sub>	"	150 days		d. -70 (21)	"	GF <sub>2</sub>	"	100 hr		e. -70 (21)	"	GF <sub>2</sub>	"	150 days		f. -70 (21)	"	GF <sub>2</sub>	"	150 days		g. AIF, 95 (35)	760 (1.02 x 10 <sup>5</sup> )	GF <sub>2</sub>	10 <sup>-3</sup> (0.133)	4 hr		h. 95% R. H.	"	"	760 (1.02 x 10 <sup>5</sup> )	"	
Initial Chamber Conditions		Back-Fill Conditions		Back-Fill																																																																			
Gas/Temperature	Pressure	Gas	Partial Pressure	Exposure Time, Maximum**																																																																			
P <sub>f</sub> (°C)	torr (n/m <sup>2</sup> )		torr (n/m <sup>2</sup> )																																																																				
a. -70 (21)	10 <sup>-6</sup> (1.33 x 10 <sup>-4</sup> )	CO <sub>2</sub>	760 (1.02 x 10 <sup>5</sup> )	To Ignition (or 1 ATM pressure)																																																																			
b. -70 (21)	10 <sup>-6</sup> (1.33 x 10 <sup>-4</sup> )	GF <sub>2</sub>	10 <sup>-3</sup> (0.133)	100 hr																																																																			
c. -70 (21)	"	CO <sub>2</sub>	"	150 days																																																																			
d. -70 (21)	"	GF <sub>2</sub>	"	100 hr																																																																			
e. -70 (21)	"	GF <sub>2</sub>	"	150 days																																																																			
f. -70 (21)	"	GF <sub>2</sub>	"	150 days																																																																			
g. AIF, 95 (35)	760 (1.02 x 10 <sup>5</sup> )	GF <sub>2</sub>	10 <sup>-3</sup> (0.133)	4 hr																																																																			
h. 95% R. H.	"	"	760 (1.02 x 10 <sup>5</sup> )	"																																																																			
Total					348																																																																		

**Legend**  
 F<sub>c</sub> Layer Compression Tests  
 F<sub>i</sub> Tensile Strength  
 IG Ignition Chamber Pressure Rise  
 DG Chamber Pressure Rise  
 VS Visual Observation  
 W Weight  
 \*\*Each outgassing measurement and product identification counted as one data point: one specimen per material tested.  
 \*\*\*Or until ignition occurs  
 \*\*\*\*Only those materials which do not ignite are continued in test sequence shown.





TABLE 3 - TEST MATRIX FOR GROUND-HOLD INSULATION MATERIALS (continued 3)

Exposure Environment					Materials  J - Beta Glass Cloth NA - Narmco 1343/7119 Adhesive GY - G-207 Adhesive PF - Polyurethane Foam, 2.0 lb/ft <sup>3</sup>	Pre-exposure Tests (2 Ea for Each Material; Specimens Weighed Before and After Exposure;* Total No. of Data Points Shown in Parentheses)	In-Situ Tests	Post-exposure Tests (2 Ea for Each Material; Specimens Weighed Before and After Exposure;* Total No. of Data Points Shown in Parentheses)	No. of Test Specimens
Exposure Environment									
8) Propellants***									
Initial Chamber Conditions		Back-Fill Conditions		Back-Fill Exposure Time Maximum***					
Gas/Temperature deg (°C)	Pressure torr (n/m <sup>2</sup> )	Gas	Maximum Partial Pressure torr (n/m <sup>2</sup> )						
a.	-70 (21)	CO <sub>2</sub>	760 (1.02 x 10 <sup>5</sup> )	To Ignition (or 1 ATM pressure)	J NA GY PF	VS(1) NA CY PF	IG(2) " "	VS(1) " "	2 2 2 2
b.	"	CF <sub>2</sub>	"	100 hr	"	"	"	W(1), F(2) W(1), F(2) W(1), F(2) W(1), F(2) p(1) W(1), F(2)	8
c.	-70 (21)	GO <sub>2</sub>	10 <sup>-3</sup> (1.33)	150 days	"	"	"	"	2
d.	"	"	"	100 hr	"	"	"	"	2
e.	"	GF <sub>2</sub>	"	150 days	"	"	"	"	2
f.	"	"	"	4 hr	"	"	"	"	8
g.	Air 85 (35) 85% R.H.	GF <sub>2</sub>	10 <sup>-3</sup> (1.33)	"	"	"	"	"	8
h.	"	"	760 (1.02 x 10 <sup>5</sup> )	"	"	"	"	"	8
TOTAL									244

Legend

F<sub>c</sub> Compression Strength  
 F<sub>t</sub> Tensile Strength  
 F<sub>s</sub> Shear Strength  
 IQ Ignition (Chamber Pressure Rise)  
 OG Outgassing Rates and Products (once each hour, first 8 hours; once every 24 hours thereafter)  
 VS Visual Observation  
 W Weight  
 p Density

\*\*Each outgassing measurement and product identification counted as one data point one specimen per material tested.  
 \*\*\*Or until ignition occurs.  
 \*\*\*\*Only those materials which do not ignite are continued in test sequence shown.







## Section 2

### TEST MATERIALS

Identification of the purchased test materials and a description of the test specimens (fabricated from the test materials) are provided here.

#### 2.1 TEST MATERIAL IDENTIFICATION

The test material (shown in Figs. 1-4) was ordered per the listing shown in Table 5 using the referenced specifications where applicable. The materials were inspected upon receipt; all material received met the required specifications. After receiving inspection, all materials were stored at  $70 \pm 2^\circ\text{F}$  ( $21 \pm 1^\circ\text{C}$ ) and  $40 \pm 4\%$  R.H. prior to the fabrication of the individual test specimens. All excess material was also stored in the controlled environment for the duration of the program.

#### 2.2 TEST COUPON PREPARATION

The test coupon geometries selected for each material are shown in Fig. 5; representative coupons cut to these dimensions are shown in Fig. 6. The above two figures include all the coupon shapes tested (except for the outgassing tests). Since surface area or weight and not shape is of primary importance for interpreting outgassing results, and the rate of outgassing varied from material to material, different size samples and sample shapes were used. For these reasons, no standard outgassing coupon shapes were used. The areas reported in the outgassing results represent the total nominal flat area of all external surfaces, not the true surface area of, for example, netting fibers.

In Volume II, the pertinent sample weight and area data are provided with the outgassing data for each material tested.

The methods used for fabricating the test coupons plus the rationale used to select a particular coupon size and geometry are summarized in Table 6.



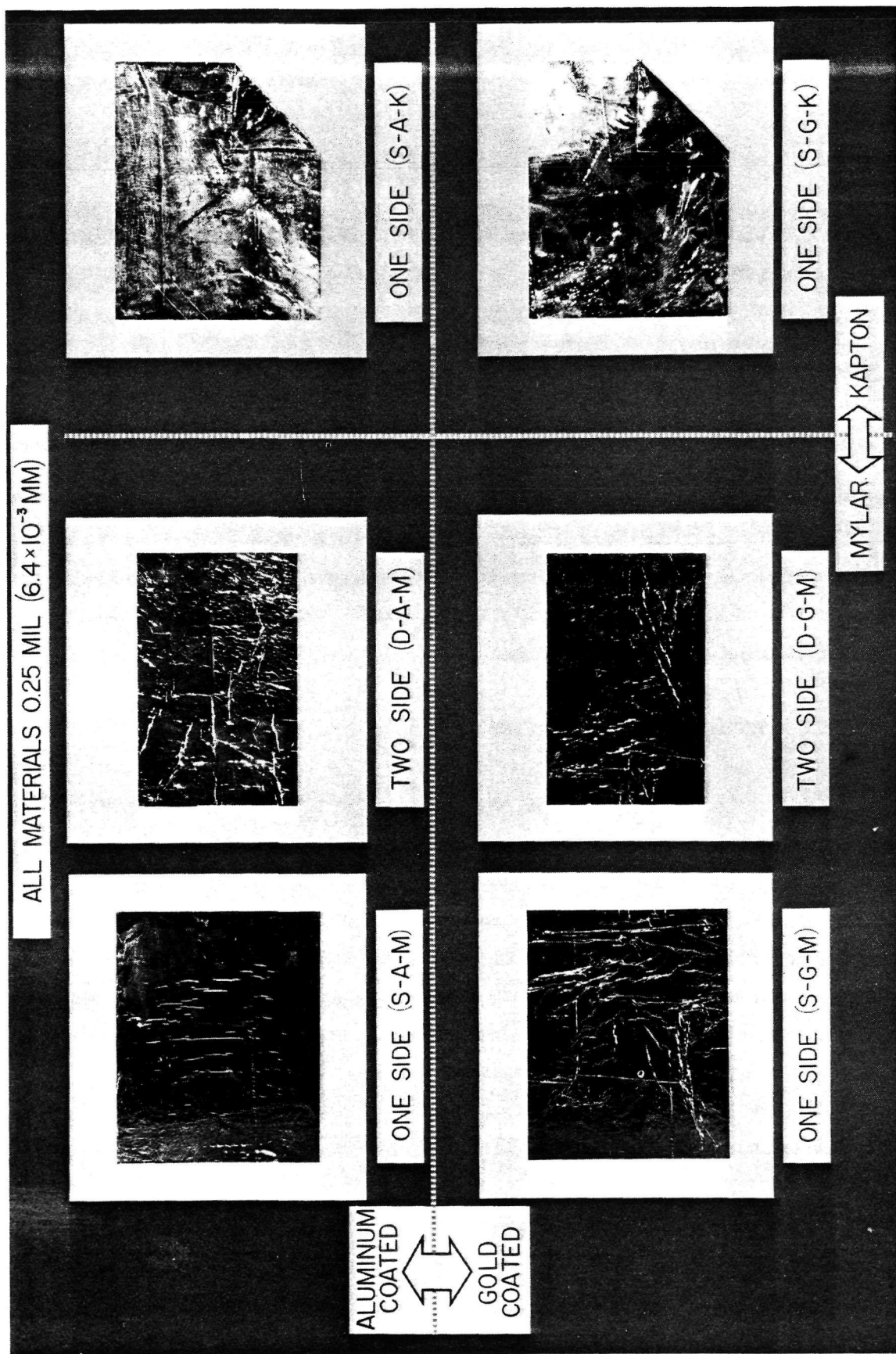


Fig. 1 Multilayer Radiation Shield Materials

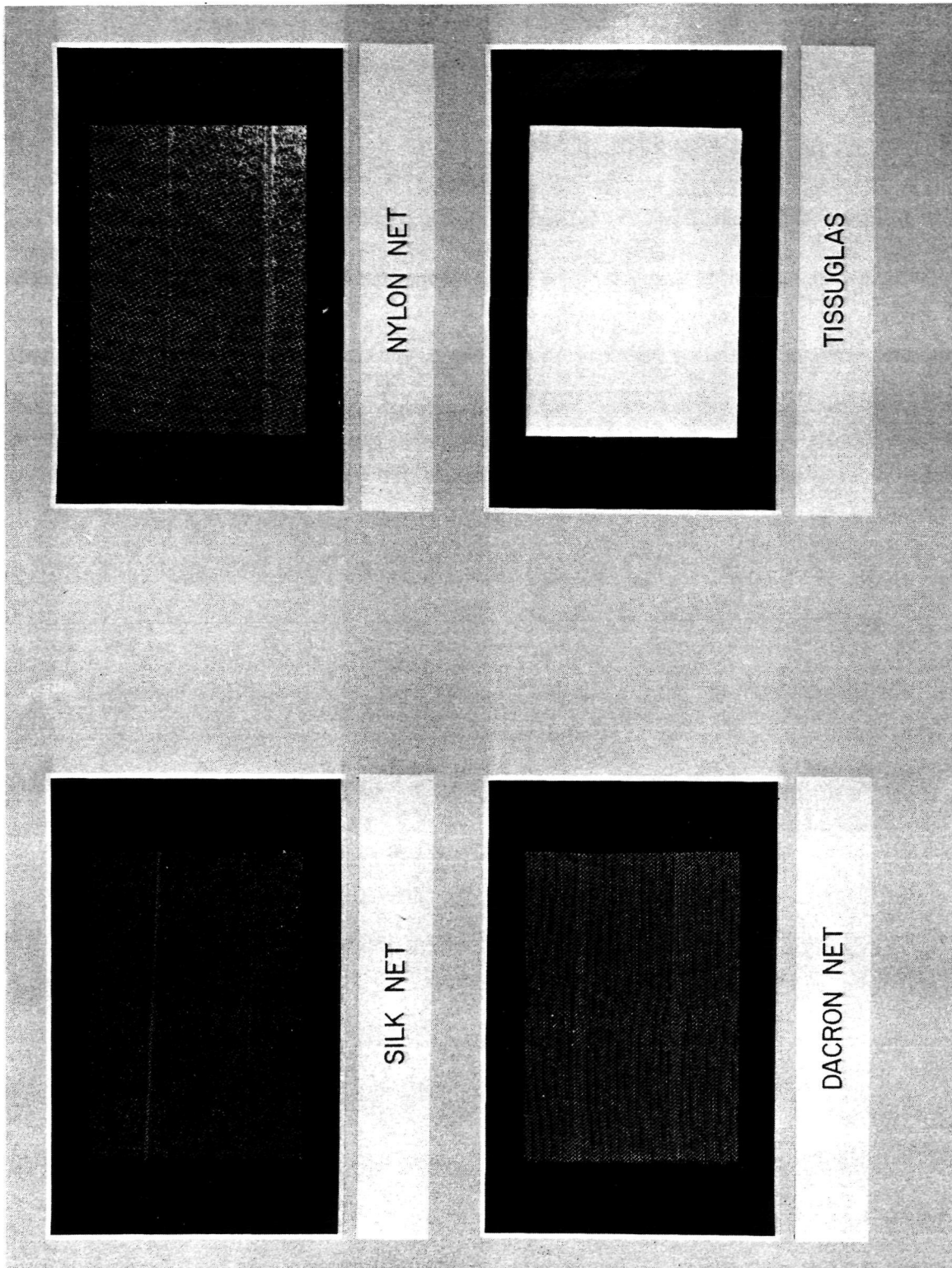


Fig. 2 Multilayer Spacer Materials

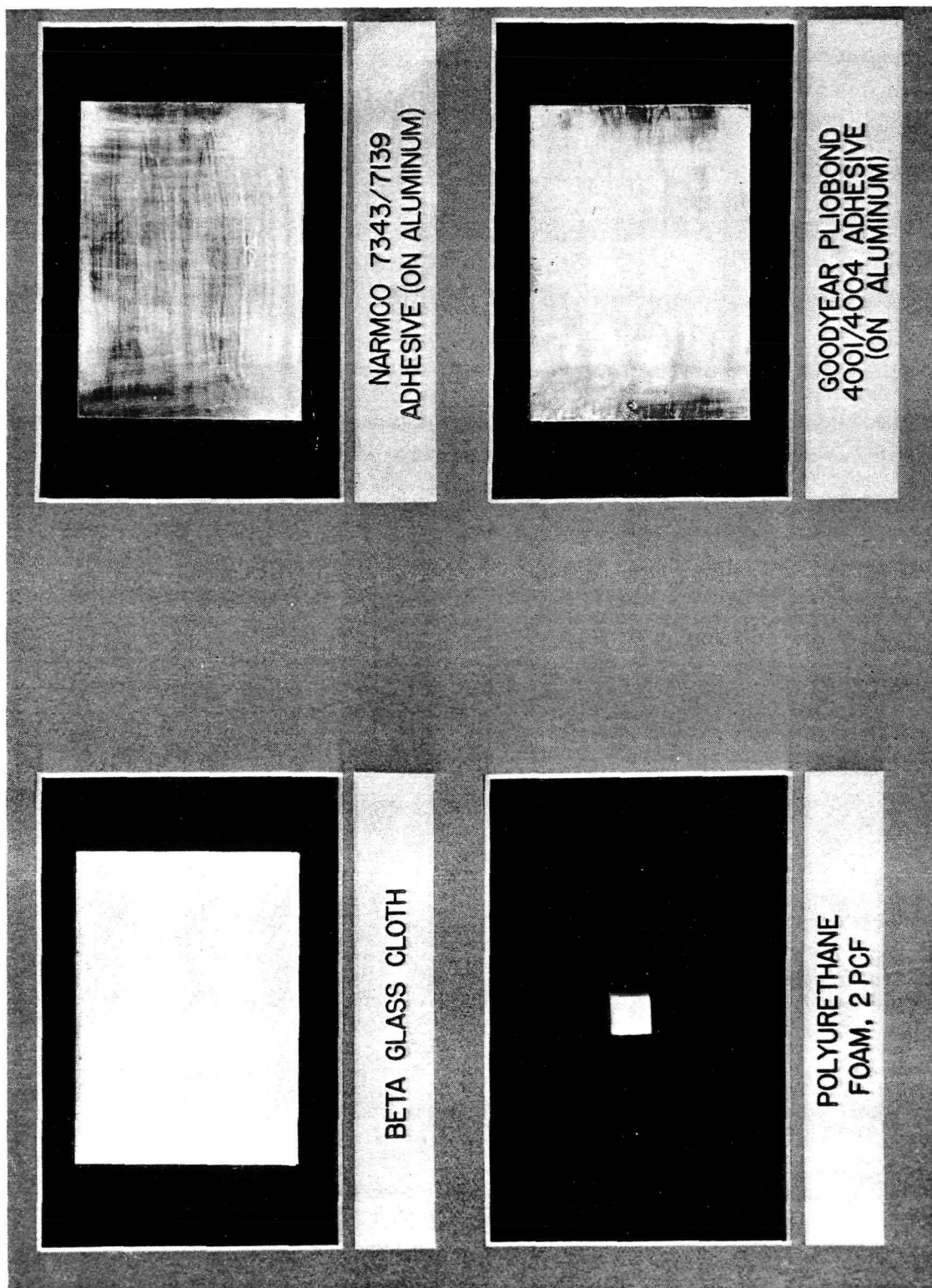


Fig. 3 Ground-Hold Insulation Materials



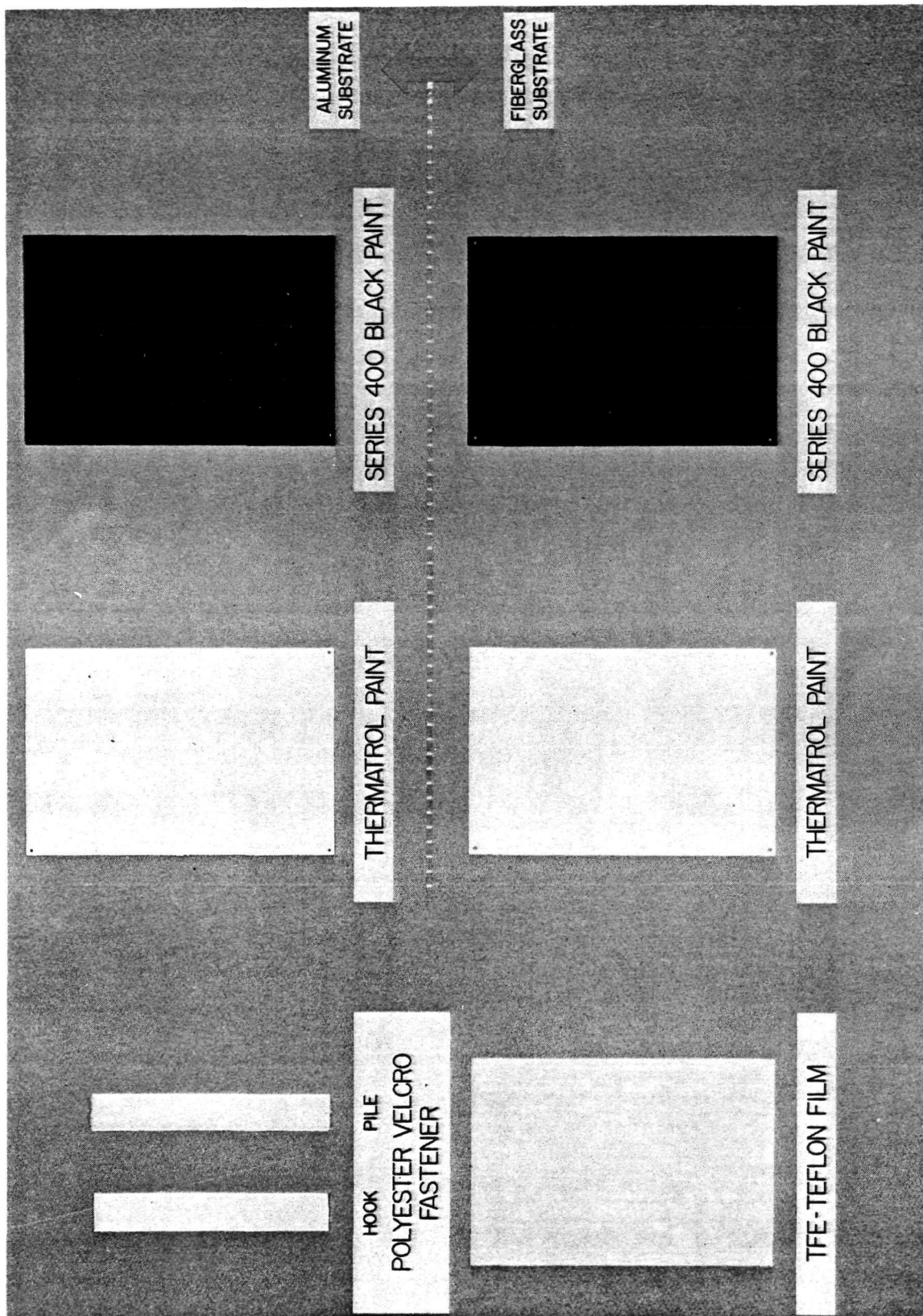


Fig. 4 Miscellaneous Insulation Materials

TABLE 5 - IDENTIFICATION OF TEST MATERIALS

## INSULATION SPACER MATERIALS

Test Material	Test Material Source	Applicable Purchase Spec.	Nominal Thickness mils (mm)
<u>Silk Net</u> "Illusion Silk Net," approx. 1/16 in. Hexagonal Mesh	John Beathcoat Company, 108 W 39th Street New York, New York 10018	None	5 (0.13)
<u>Nylon Net</u> 1/16 x 3/32 in. Hexagonal Mesh	Sears Roebuck & Co. (Any local store)	None	9 (0.23)
<u>Tissauglas</u> Style 600	Phillips Products Corp., Kennedy Drive Putnam, Connecticut 06860	LAC 26-4392	0.6 (1.52x10 <sup>-3</sup> )
<u>Dacron Net</u> (.0028 lb/ft <sup>2</sup> ) 3/24, 1962 meshes/in. <sup>2</sup>	Apex Mills 49 W. 37th Street New York, New York 10018	None	6.7 (0.12)

## RADIATION SHIELD MATERIALS

Test Material	No. of Sides Coated	Test Material Source	Applicable Purchase Spec.
<u>Film Coating</u>			
<u>Mylar-Aluminum</u>	1	National Metallizing Div., Standard Packaging Corp., Cranbury, New Jersey 08512	LAC 22-4402
<u>Mylar-Aluminum</u>	2		LAC 22-4402
<u>Kapton-Aluminum</u>	1		LAC 22-4413
<u>Mylar-Gold</u>	1		LAC 22-4402 + Addendum A
<u>Mylar-Gold</u>	2		LAC 22-4402 + Addendum A
<u>Kapton-Gold</u>	1		LAC-4413 + Addendum A

\* Nominal thickness 0.25 mils (6.4 x 10<sup>-3</sup> mm)

## MISCELLANEOUS INSULATION MATERIALS

Test Material	Test Material Source	Applicable Purchase Spec.	Nominal Thickness mils (mm)
<u>Velcro Fasteners</u> 100% Polyester 1-in. wide, white RFS-12-1-100 Hook RFS-12-1-100 Loop	The Hartwell Corporation 9035 Venice Blvd Los Angeles, Ca 90034	None	-
<u>Teflon Film</u> TFE	E. I. du Pont, de Nemours & Co., Inc., Wilmington, Del 19898	AMS 3651 RFS 22-302	10 (0.25)
<u>Thermalol Paint</u> (24-100)	Lockheed Missiles & Space Co. (LMSC) Sunnyvale, Ca 94088	LAC 37-4094 101 (Lockheed Process Bulletin 55)	-
<u>3M Co. Series 400 Black Paint</u> with Primer, DuPont 65-3011, Dark Gray	Reflective Products Division 3M Co., 3M Center, St. Paul, Minnesota 55101	None	-
<u>Paint Substrate</u> Epoxy Glass	LMSC	ML-P-18177C	16 (0.41)
<u>Aluminum, 6061-T6</u>	LMSC		20 (0.51)

## GROUND HOLD INSULATION MATERIALS

Test Material	Test Material Source
<u>Beta Fiber Fabric</u> (6.3 oz/yd) Style 19035 Finish 9362	J. P. Stevens & Co., Inc. 1185 Avenue of the Americas New York, New York 10036
<u>Maroco 7343/7139 Adhesive</u>	Maroco Materials Div. Victoria & Placentia Streets Costa Mesa, California 92627
<u>Goodyear Pliobond Adhesive</u> 4001/4004	Goodyear Tire & Rubber Company Adhesive Dept. 1745 Cottage Street Ashland, Ohio 44805
<u>Polyurethane Foam</u> (2 lb/ft <sup>3</sup> ), MC-250-A	Kopco Chemical Division of Diamond Shamrock Corp. 60 Park Place Newark, New Jersey 07101 (Blown foam supplied by North American Rockwell Corp.)

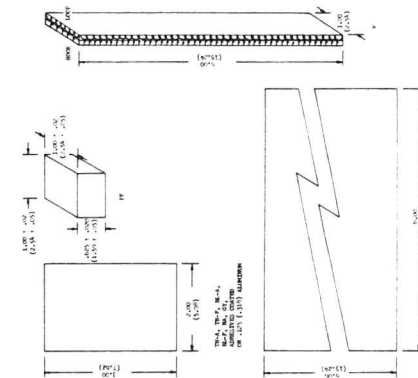
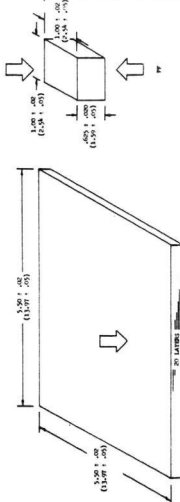
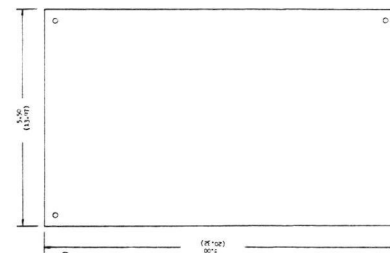
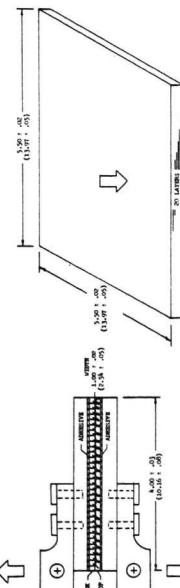


Fig. 5 Design of the Test Coupons

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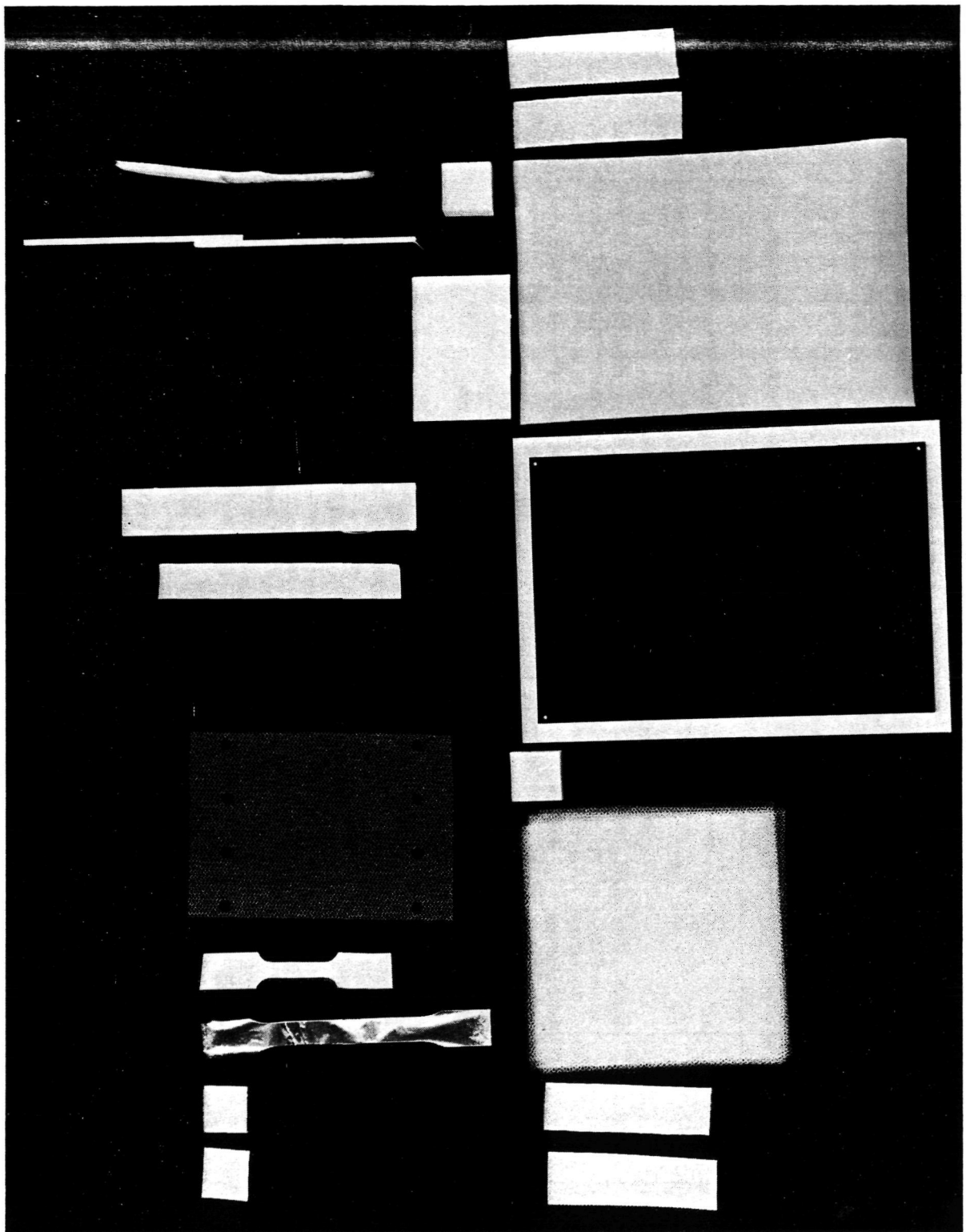


Fig. 6 Representative Test Coupons

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TABLE 6  
SUMMARY OF COUPON FABRICATION METHODS AND RATIONALE FOR SELECTION OF PARTICULAR COUPON GEOMETRIES

COUPON	MATERIAL	FABRICATION METHOD	RATIONALE FOR SELECTION OF COUPON GEOMETRIES
Tensile	S-A-M, D-A-M, S-A-K, S-G-M, D-G-M, S-G-K	Metal templates, scalpel	Prior tests on 1/4 mil films yielded consistent repeatable data A rectangular shape was chosen over the "dog-bone" shape because strands terminating in the curved section of the dog-bone are not anchored into the system. The width of the ungripped portion of the rectangular pattern was set greater than the length to minimize "puckering" or "necking-down" and is more representative of nets used in large sheets. Even with a rectangular pattern, netting nodal points do not always fall on a straight line thus changing the number of mesh strands over the coupon length. This characteristic of nets tends to widen the test scatter band but a rectangular shape is still a better choice than the "dog-bone" shape since the same problem occurs in the straight test section for any of the coupons.
	NN, DN, SN	Metal templates, scalpel (see Fig. 6 for orientation of netting with respect to stress axis)	The tissuglas was too delicate to be tested in the same fashion as the net. It was found that narrow rectangular strips were less prone to damage in handling, and easier to install in the tester. The slightest wrinkle causes breaking. The narrow specimens are less prone to wrinkling. The Beta-glass cloth had to be treated as a special case. Its vastly greater strength made it necessary to use a thin-rectangular strip and switch to a different testing machine. The specimen is rectangular for the same reason as the net samples.
	TG	Metal template, scalpel	
	$\beta$	Metal templates, scalpel	
	V	Metal rule, scalpel. Bond Velcro to aluminum blocks with Epibond I23/50 PBW 9615 hardener adhesive after environmental exposure	Standard width of Velcro used in insulation systems. Bonding performed after exposure to prevent possible Velcro/adhesive interaction in the environment.

CONTINUATION OF  
SUMMARY OF COUPON FABRICATION METHODS AND RATIONALE FOR SELECTION OF PARTICULAR COUPON GEOMETRIES

Tensile	TFE	Stacked film, metal templates on each side; machine coupon shape.	Extension of TFE 300% to 500% during test required a smaller test specimen than the radiation shields so that cross-head travel of the tensile machine was not exceeded.
Tensile Shear	NA	Degrease adherend surface with MEK. Hand-sand adherend with W-400A dry abrasive paper. Remove all loose particles & re-clean with MEK. Air dry 15 minutes. Melt 100 PBW of 7139 at 250°±25°F (121 ± 14°C). Stir into 7343 at room temperature. Squeegee ~5 mils of adhesive on prepared adherend surfaces. Clamp together for 3 days at 70°F (21°C).	Aluminum adherends simulate the cryogenic tank surface. The bonding procedure is per the manufacturer's recommendations.
	GY	Prepare adherend surfaces as described above. Mix 4 PBW of 4004 with 100 PFW of 4001. Squeegee ~5 mils of adhesive on prepared adherends. Dry using infrared lamps. Activate film at 230-250°F (110 to 121°C). Join adherends together while adhesive is tacky. Clamp together for >24 hours at room temperature.	Aluminum adherends simulate the cryogenic tank surface. The bonding procedure is per the manufacturer's recommendations.
	V	Metal rule, scalpel. Bond Velcro hook portion to aluminum adherend with Epibond I23/50 PBW 9615 Hardener adhesive after environmental exposure.	Standard width of Velcro used in insulation systems. Bonding performed after environmental exposure to prevent possible Velcro/adhesive interaction in the environment.

CONTINUATION OF  
SUMMARY OF COUPON FABRICATION METHODS AND RATIONALE FOR SELECTION OF PARTICULAR COUPON GEOMETRIES

Compression ↓	NM, DN, SN, TG PF	Metal template, scalpel	Spacer area and number are large enough to be representative of an installed system.
Peel	V	Metal guide plates, scalpel	Thickness tested is representative of installed sublayer insulation system.
		Metal rule, scalpel	T-peel test fixture has produced reproducible results in prior test programs.
Adhesion, Flexibility, Solar Absorptance, Solar Transmittance, Emittance ↓	S-A-M, D-A-M, S-A-K, S-G-M, D-G-M, S-G-K TH-A, TH-F BL-A	Metal template, scalpel	Adequate size to make required measurements.
		Prepare per Lockheed PB-105-101	Adequate size to make required measurements. Coated both sides and edges to prevent substrate exposure to the environments.
		Clean metal surface with MEK. Prime surface with DuPont 65 Line Preparation Primer Surface; air dry for 24 hours. Mix 2 components per manufacturer's recommendations and apply using standard spray painting procedures. Air dry for > 24 hours.	Adequate size to make required measurements. Coated both sides and edges to prevent substrate exposure to the environments.
	BL-F	Omit primer coat. Application same as above.	Adequate size to make required measurements. Coated both sides and edges to prevent substrate exposure to the environments.
Ignition ↓	S-A-M, D-A-M, S-A-K, S-G-M, D-G-M, S-G-K, NM, DN, SN, TG, $\beta$ , PF, V, TFE NA, GY TH-A, TH-F, BL-A, BL-F	Metal guides, scalpel	Sufficient surface area for ignition (engineering estimate)
		Spatula to spread adhesive on aluminum plate (see tensile shear tests for cure cycle)	Sufficient surface area for ignition (engineering estimate)
		See Adhesion, Flexibility procedure listed above	Sufficient surface area for ignition (engineering estimate)

LEGEND

MATERIALS

S-A-M	Single, Aluminized Mylar	$\beta$	Beta Glass Cloth
D-A-M	Double, Aluminized Mylar	NA	Narmco 7343/7139 Adhesive
S-A-K	Single, Aluminized Kapton	GY	Goodyear 4001/4004 Adhesive
S-G-M	Single, Goldized Mylar	PF	Polyurethane Foam
D-G-M	Double, Goldized Mylar	V	Velcro Fasteners
S-G-K	Single, Goldized Kapton	TFE	Teflon
SN	Silk Net	TH-A	Thermatrol on Aluminum
NN	Nylon Net	TH-F	Thermatrol on Fiberglass
DN	Dacron Net	BL-A	3M Black Paint on Aluminum
TG	Tissuglas	BL-F	3M Black Paint on Fiberglass

### 2.3 TEST COUPON IDENTIFICATION

All test specimens were assigned a sample number and identified using an imprinted aluminum tag. The tag and sample were stored in polyethylene bags prior to and following the exposures. In general, no markings were made on the specimens (due to possible reaction of the marking material in the exposure environment). Exceptions to this rule are the adhesives and paints where markings were scribed into the materials in areas where the test results were not affected.

For specimens where each side had to be identified; i.e., radiation shields and thermal control paints, holes were punched in three corners of the rectangle as shown previously in Fig. 5. This pattern on a rectangular surface uniquely identifies each side.

Identification of the materials in the environmental exposure chambers were maintained by "mapping" the chamber or use of the aluminum tag on samples where it could be attached.

### 2.4 SUPPORT OF THE TEST COUPONS IN THE ENVIRONMENTAL EXPOSURE CHAMBERS

Stainless steel springs and wires were used to suspend the test specimens in the various environmental exposure chambers. The 0.5 in (1.27 cm) diameter spring was cut into approximately .25 in. (.635 cm) sections. The springs were expanded and slipped over two adjacent corners of the test specimens. Stainless steel wires, attached to the two springs were used to individually support each coupon from racks or support bars available in the environmental chambers.

Some of the test coupons, such as the paint samples had identification holes punched in the corners through which the stainless steel wires were attached. Each sample was carefully hung in the vertical position and separated from the other samples so all faces of the coupon were exposed to the environmental conditions to which it was being subjected.

### Section 3

#### ENVIRONMENTAL EXPOSURE CONDITIONS

Pre-exposure measurements as called out in Tables 1-4 were made on the test coupons, the coupons were then exposed to eight environmental exposure conditions (including a control environment for reference) and post-exposure measurements were made within one working day after removal of the coupons from the environment. For two of the properties measured - outgassing in environments 2b, 2c, 2d and 4 and possible ignition upon  $O_2$  or  $F_2$  exposure in environments 8a and 8b - the data were obtained during the environmental exposure. Special environmental exposure equipment was constructed for environments 2, 4 and 8. Details on the environmental exposure conditions are provided below in the same sequence provided previously in Tables 1-4.

##### 3.1 CONTROL (Environment 1)

All test coupons (in marked polyethylene bags) were stored in an automatically controlled constant temperature  $70 \pm 2^\circ F$  ( $21 \pm 1^\circ C$ ), constant humidity  $40 \pm 5\%$  room prior to their respective environmental exposures. In addition, property data were obtained on the "as-received" material on 1 November 1970 and after  $10 \pm 3$  days and  $150 \pm 5$  days in the controlled environment given above. Continuous records of temperature and humidity verified the ranges shown. The data obtained at 0, 10 and 150 days were used as a reference standard against which the environmental exposure data could be compared (as discussed in Section 6.0).

##### 3.2 VACUUM (Environment 2)

Test coupons were maintained at temperatures of 660, 530, 140 and  $37^\circ R$  (366, 294, 78 and  $21^\circ K$ , respectively) at a pressure of  $\leq 10^{-6}$  torr ( $1.33 \times 10^{-4}$  n/m<sup>2</sup>) for periods up to 240 hours after which various mechanical and physical

property tests were performed. In-situ outgassing tests were also conducted at all the above listed temperatures except 37°R (21°K).

As the intent of this study was to obtain long term vacuum exposure data, all specimens were tested for the maximum time, 240 hours. Certain materials were also exposed for 24 hours at 660°R (366°K) to assess the effect of exposure time.

Vacuum exposures at 660°R (366°K) and 530°R (294°K) were performed in a vacuum chamber at  $\leq 10^{-6}$  torr ( $\leq 1.33 \times 10^{-4}$  n/m<sup>2</sup>). A compartmented, copper box was placed in the vacuum chamber as shown in Fig. 7. Specimens were suspended on racks in any one or all of the six compartments. A uniform temperature was maintained in the box with heater elements located on all sides of the box. A separate element was used for the lid for ease of installation and removal of the test specimens. The temperature was maintained to  $\pm 9^\circ\text{R}$  (5°K) and monitored with Cu-Co thermocouples, accurate to  $\pm 4^\circ\text{R}$  (2°K).

Vacuum exposures at 140°R (78°K) and 37°R (21°K) were performed in the test setup shown in Fig. 8. The test specimens were placed in holding racks within the pie-shaped compartments of the double-walled cylindrical container. A copper lid was placed over the top, the overall chamber was pumped down to  $\leq 10^{-6}$  torr ( $1.33 \times 10^{-4}$  n/m<sup>2</sup>) and the inverted U-shaped outer guard was filled with LN<sub>2</sub>. (Time to pressure varied from 1 to 4 hours depending on the test material.) The double-walled container was then filled with LN<sub>2</sub> or LH<sub>2</sub> depending on the exposure temperature desired.

Sample temperatures were assumed to be in equilibrium with the inner cryogen vessel temperature. This temperature was determined by measurement of pressure in the cryogen vessel ( $\pm .9^\circ\text{R}$  or  $\pm .5^\circ\text{K}$ ). At the conclusion of this period, the cryogen vessels were emptied and the chamber brought to and maintained at atmospheric pressure with a dry He purge. Purging assures uniform temperature during warmup. When the specimen reached  $> 513^\circ\text{R}$  ( $> 285^\circ\text{K}$ ) (as determined



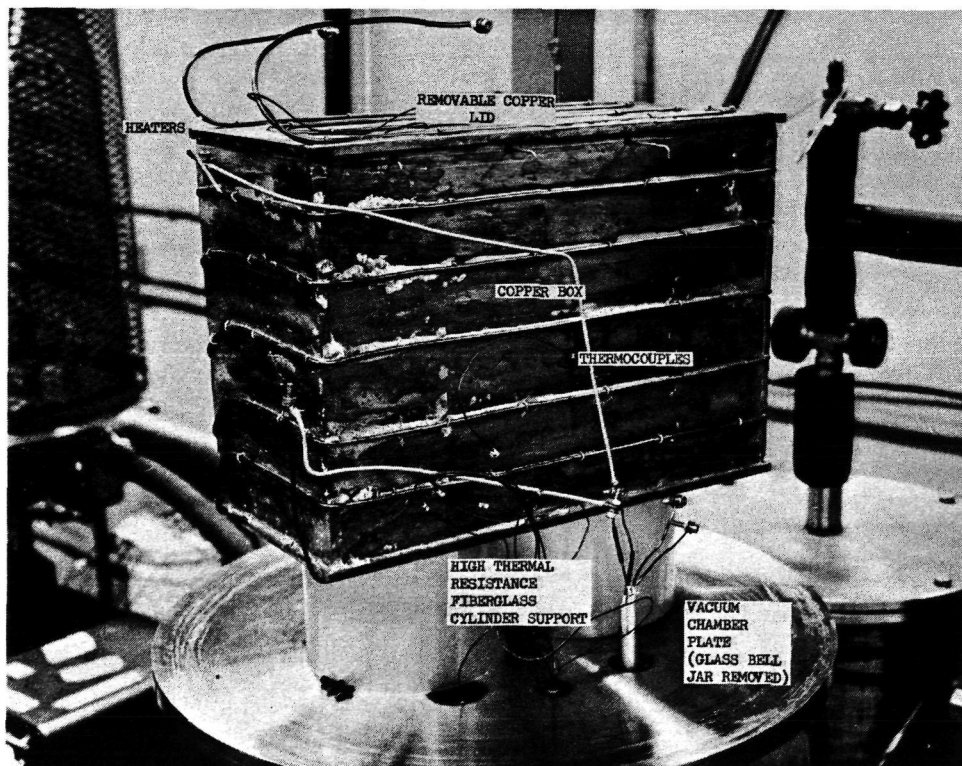
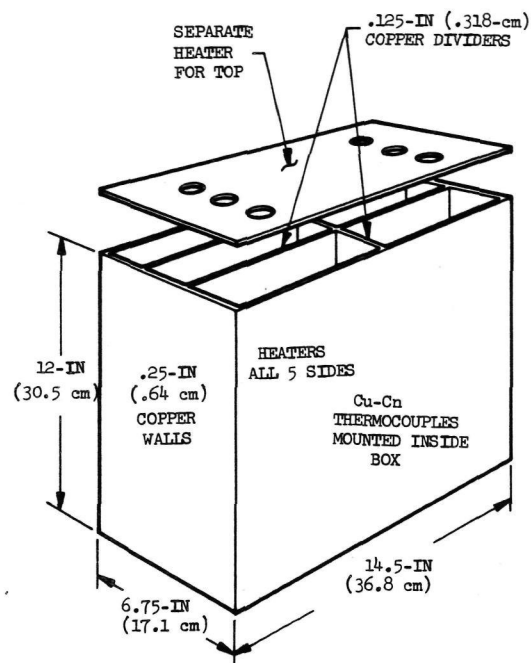


Fig. 7 High Temperature Vacuum Exposure Apparatus  
(Environments 2a, 2b and 2c)

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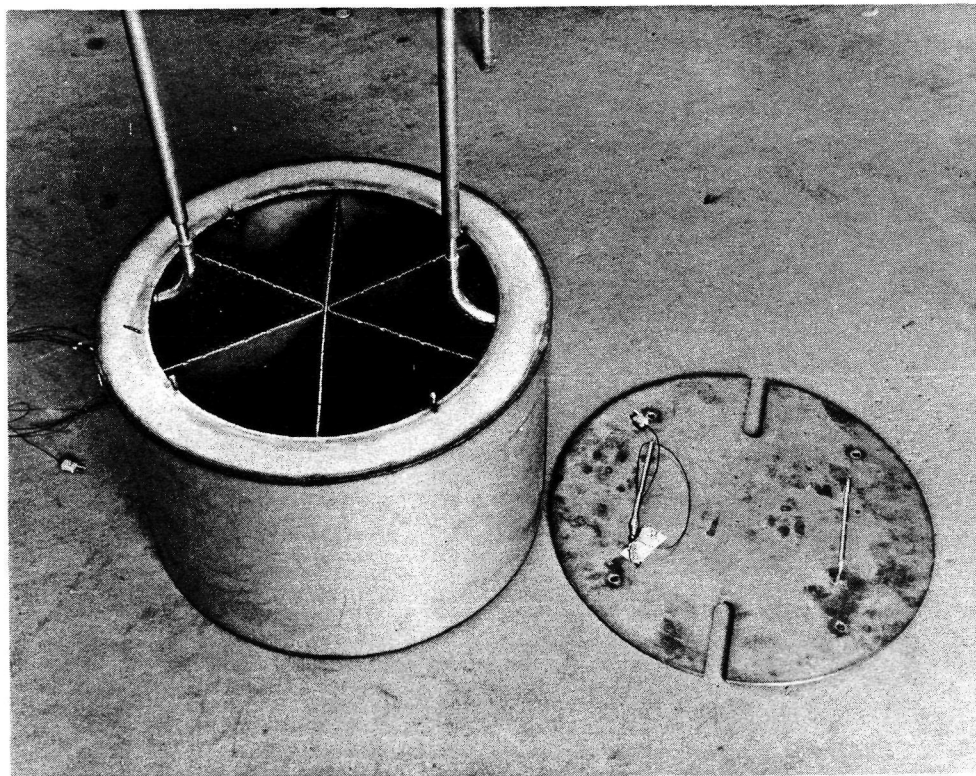
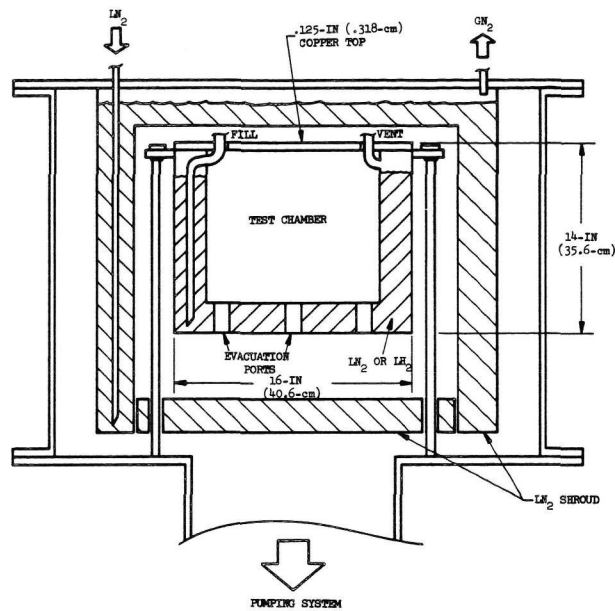


Fig. 8 Low Temperature Vacuum Exposure Apparatus  
(Environments 2d and 2e)

by thermocouple instrumentation on the inner chamber wall) a temperature where moisture condensation will not occur, the vacuum chamber was opened and the specimens removed for property testing. The specimen warmup period was on the order of 8 hours for this environment.

An atmosphere of He was maintained in the chamber when not in use.

### 3.3 HIGH TEMPERATURE (Environment 3)

The test materials were exposed to an air atmosphere at 200°F (93°C) and 40 percent relative humidity for periods of 24, 72, and 240 hours.

A forced convection laboratory oven (Tunney Model TH 270200) with an automatically controlled atomizing-type humidifier (steam from distilled water source) was used. Temperature uniformity within the oven is  $\pm 5^\circ\text{F}$  ( $3^\circ\text{C}$ ) and relative humidity control is  $\pm 4$  percent. Specimens were suspended from racks with adequate separation for uniform exposure of all surfaces as shown in Fig. 9. Continuous records of temperature (wet and dry bulb) were maintained throughout the test period.

The specimens were suspended from the shelves and the oven brought up to operating conditions within 30 minutes. At the conclusion of the exposure period, the oven was shutdown and purged with dry He at 70°F (21°C) to cool the specimen to ambient condition before removal.

### 3.4 VACUUM - PURGE - VACUUM (Environment 4)

This environment was used only for the outgassing tests and is discussed in Section 4.11.

### 3.5 95% HUMIDITY (Environment 5)

The test materials were exposed to an air atmosphere of 95°F (21°C) and 95%



Fig. 9 High Temperature (Environment 3) and 95% Humidity (Environment 5) Exposure Chamber

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relative humidity for periods of 12, 24 and 72 hours. The specimens were maintained at  $95 \pm 4^{\circ}\text{F}$  ( $35 \pm 2^{\circ}\text{C}$ ) in air at a relative humidity of  $95 \pm \frac{5}{0}$  percent relative humidity environment in the same forced convection oven used in environment 3. The chamber is equipped with automatic temperature and humidity controls and instrumentation to provide a continuous record of both temperature and relative humidity.

Specimens were suspended from racks and the chamber was brought to environmental operating conditions within 10 minutes. At the conclusion of the test, the chamber temperature was reduced to  $70^{\circ}\text{F}$  ( $21^{\circ}\text{C}$ ) and the humidity held at 80/95 percent prior to specimen removal.

### 3.6 95% HUMIDITY - SALT AIR (Environment 6)

All materials were exposed to a salt air environment at  $95 \pm 4^{\circ}\text{F}$  ( $35 \pm 2^{\circ}\text{C}$ ) and  $95 \pm \frac{5}{0}$  percent relative humidity for periods of 12, 24, and 72 hours.

This environmental condition was maintained in a Conrad Salt Spray Chamber Model T-CA-1. The working volume is 17.5 cu. ft. (33-in. H x 25-in. W x 37-in. L). This chamber provides the required conditions in accordance with MIL-E-5272001.

Specimens were suspended from bars with adequate separation for uniform exposure of all surfaces as shown in Fig. 10. The chamber was brought to operating conditions in minutes and held there for the specified time. At the conclusion of the test, the chamber temperature was reduced to  $70^{\circ}\text{F}$  ( $21^{\circ}\text{C}$ ) and the humidity held at 80/95 percent prior to specimen removal.

### 3.7 WATER IMMERSION (Environment 7)

Cleaned pyrex beakers were filled with distilled water and held at  $70 \pm 5^{\circ}\text{F}$  ( $21 \pm 3^{\circ}\text{C}$ ). Specimens were totally immersed in the water for periods of 0.5, 2 and 24 hours. Racks kept the materials separated and immersed and

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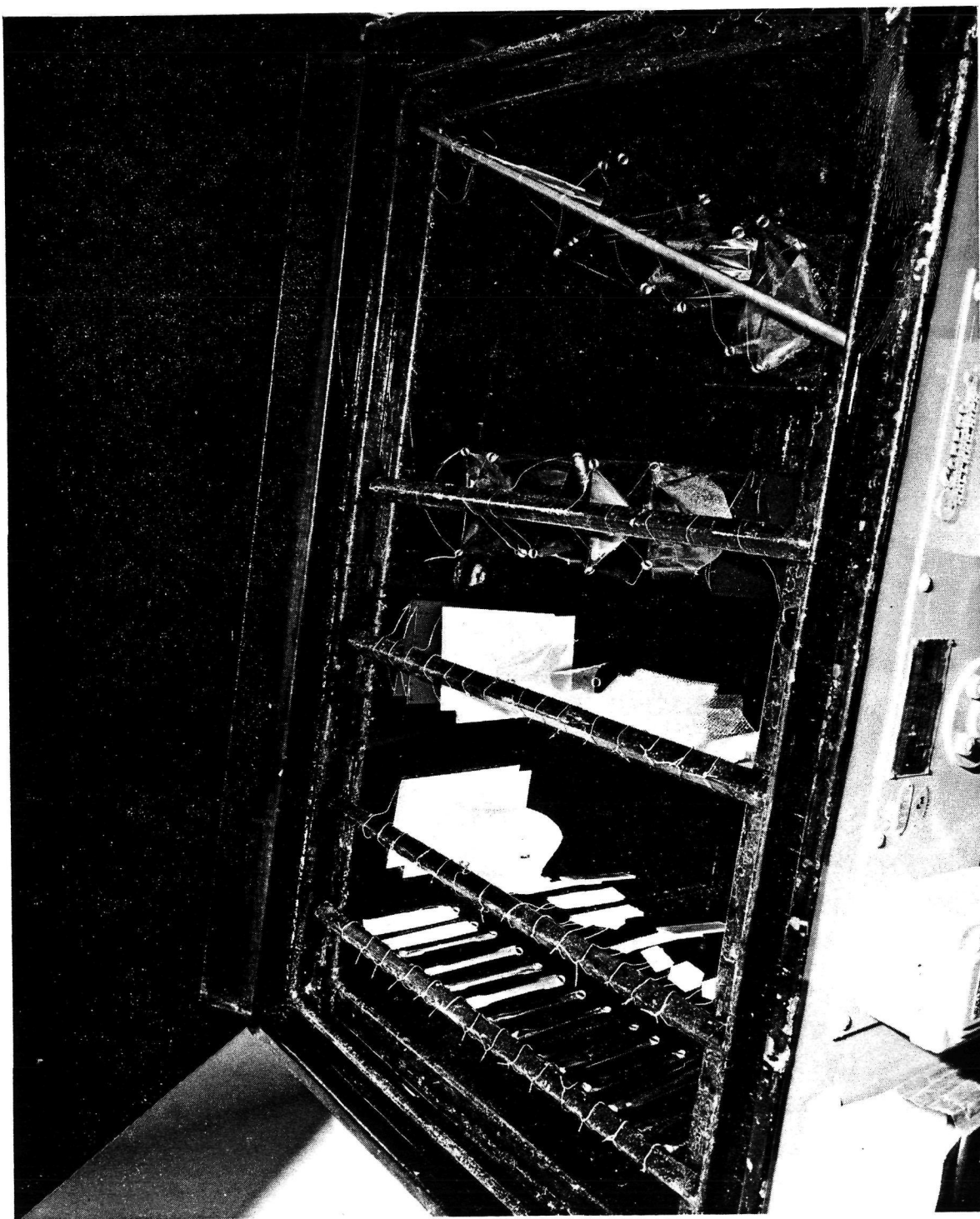


Fig. 10 95% Humidity/Salt Air Exposure Chamber (Environment 6)

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prevented "floating" of the material. At the conclusion of the immersion period, the specimens were air dried in the control environment (Environment 1) prior to post-exposure property measurements.

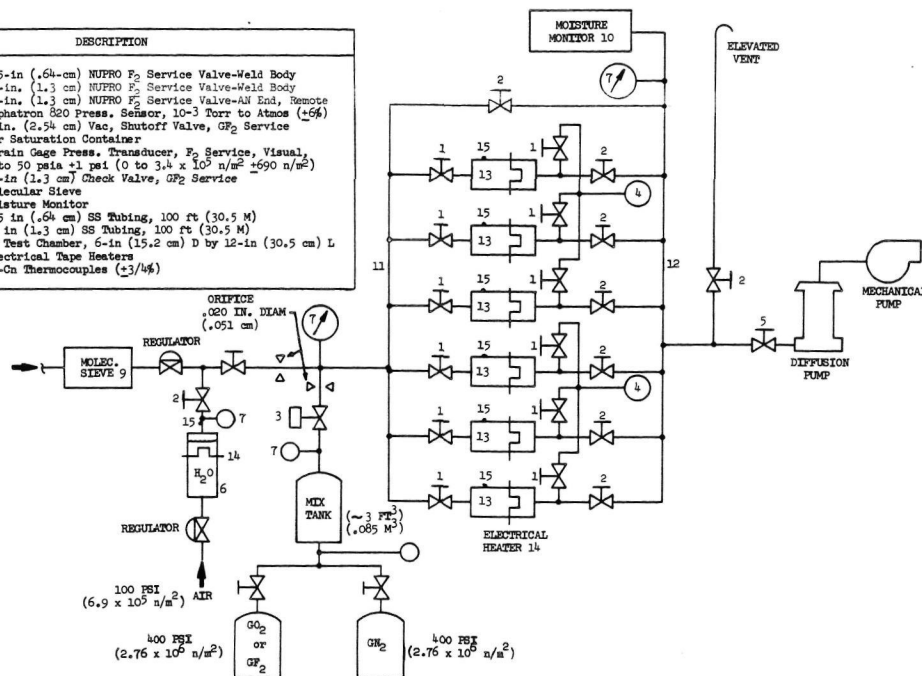
### 3.8 GASEOUS PROPELLANT EXPOSURE (Environment 8)

Environmental exposure testing was conducted in three phases using gaseous  $O_2$  and  $F_2$ . In the first phase (Environments 8a and 8b) the specimens were placed in a reaction chamber, pressure was reduced to  $10^{-6}$  torr ( $1.33 \times 10^{-4}$  n/m<sup>2</sup>) and then back-filled with the dry gaseous propellant until a reaction was detected by chamber pressure discontinuity or until a pressure of one atmosphere was achieved without evidence of reaction. In the second phase (Environments 8c, 8d, 8e and 8f) the specimens were placed into a chamber which was evacuated to a pressure of  $10^{-6}$  torr ( $1.33 \times 10^{-4}$  n/m<sup>2</sup>) and then back-filled with the dry gaseous propellant to a pressure of  $10^{-3}$  torr (.133 n/m<sup>2</sup>). Specimens were maintained at the pressure for periods of 100 hours and 150 days or until a reaction was detected by a chamber pressure rise. Only those materials which did not show a reaction at a pressure of  $10^{-3}$  torr (.133 n/m<sup>2</sup>) or less in the initial phase was tested in this second phase. The third phase (Environments 8g and 8h) consisted of exposing the specimens to a mixture of air at  $95 \pm 4^\circ F$  ( $35 \pm 2^\circ C$ ) and relative humidity of  $95 \pm 5\%$  with  $GF_2$ , first at a partial pressure of  $10^{-3}$  torr (.133 n/m<sup>2</sup>) and secondly at a partial pressure of one atmosphere. Exposure times were a maximum of four hours.

The test equipment and system used for the vacuum/propellant exposure testing is shown in Fig. 11. Fig. 12 shows test specimens suspended inside a propellant exposure chamber. Six (6) test chambers were used for the testing as shown in Fig. 11 for the long term (150 days) exposure to eliminate the possibility of the reaction of one type of specimen affecting another type of specimen.

The procedure used for performing the tests was as follows. (Refer to Fig. 11 and the numbering nomenclature used.)

ITEM NO.	DESCRIPTION
1	.25-in (.64-cm) NUPRO F <sub>2</sub> Service Valve-Weld Body
2	.5-in. (1.3 cm) NUPRO F <sub>2</sub> Service Valve-Weld Body
3	.5-in. (1.3 cm) NUPRO F <sub>2</sub> Service Valve-Ad End, Remote
4	Alphatron 820 Press. Sensor, 10-3 Torr to Atmos ( $\pm 6\%$ )
5	1-in. (2.54 cm) Vac. Shutoff Valve, GP <sub>2</sub> Service
6	Air Saturation Container
7	Strain Gage Press. Transducer, F <sub>2</sub> Service, Visual, 0 to 50 psia $\pm 1$ psi (0 to $3.4 \times 10^5$ n/m <sup>2</sup> $\pm 690$ n/m <sup>2</sup> )
8	.5-in (1.3 cm) Check Valve, GP <sub>2</sub> Service
9	Molecular Sieve
10	Moisture Monitor
11	.25 in (.64 cm) SS Tubing, 100 ft (30.5 M)
12	.5 in (1.3 cm) SS Tubing, 100 ft (30.5 M)
13	SS Test Chamber, 6-in (15.2 cm) D by 12-in (30.5 cm) L
14	Electrical Tape Heaters
15	Cu-Cn Thermocouples ( $\pm 3/4\%$ )



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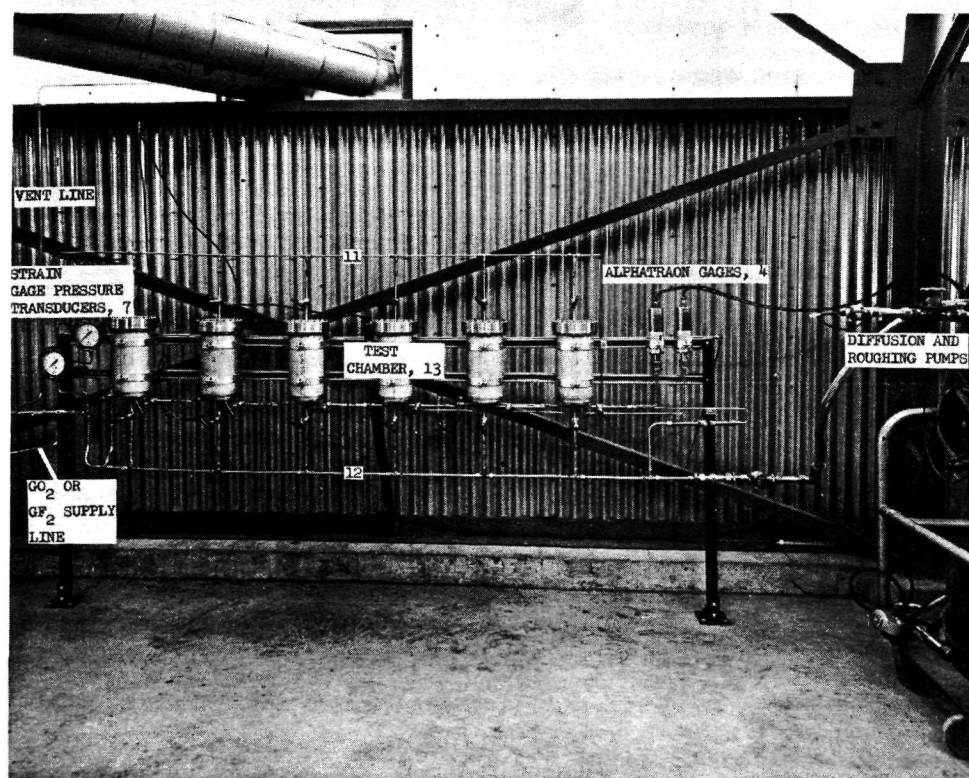


Fig. 11 Gaseous Propellant Exposure Apparatus  
(Environment 8)

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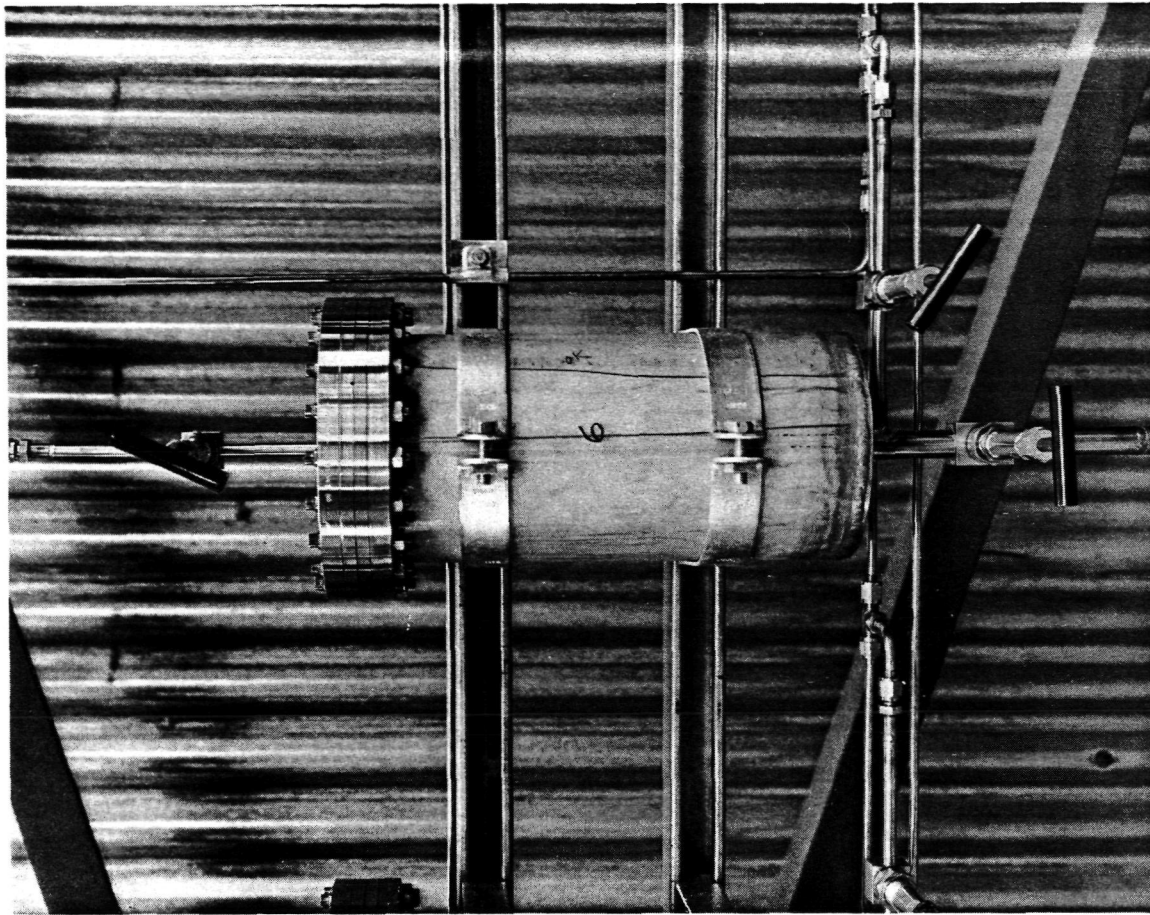
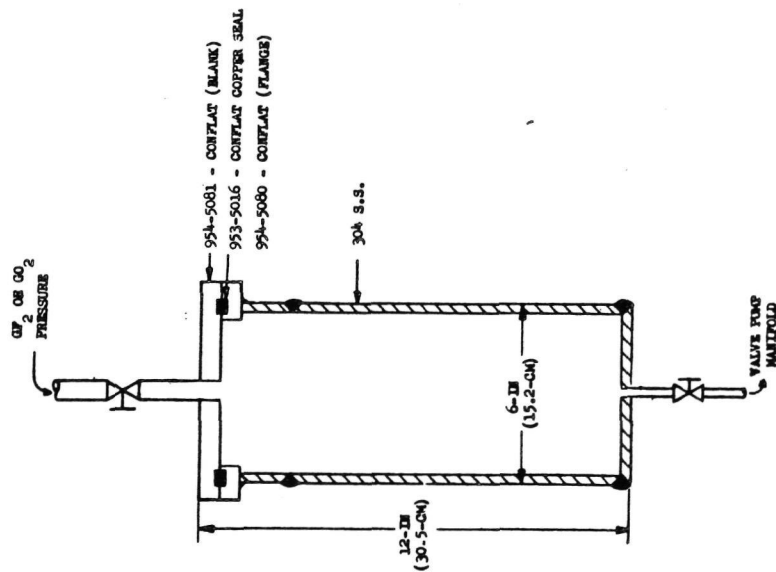


Fig. 12 Gaseous Propellant Exposure Chamber (Environment 8)



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Environments 8a and 8b. Test specimens mounted on racks were installed and exposed in each test chamber, 13, to a mean ambient temperature of  $68 \pm 18^\circ\text{F}$  ( $20 \pm 10^\circ\text{C}$ ). The chambers were pumped down to  $10^{-6}$  torr, ( $1.33 \times 10^{-4}$  n/m<sup>2</sup>), and  $\text{GO}_2$  or  $\text{GF}_2$  was introduced at the approximate rate of 0.5 psi/min (3350 n/m<sup>2</sup> per min) into one chamber at a time. The pressure response was noted on gage 7. The test was stopped when a pressure spike occurred or when one atmosphere pressure was reached. Lines 11 and 12 were then purged with  $\text{GN}_2$  between the tests. Following tests in all the chambers, the entire system was purged with  $\text{GN}_2$ .

Environments 8c, 8d, 8e and 8f. The above listed evacuation procedure was repeated. The evacuated chambers were back-filled with  $\text{GO}_2$  or  $\text{GF}_2$  and held at  $10^{-3}$  torr ( $.133$  n/m<sup>2</sup>) as noted by gage 4 for 100 hours. Pressures were monitored hourly the first 8 hours and once a day thereafter. The test was concluded when the maximum test duration was reached. A new batch of test materials is then exposed to  $\text{GO}_2$  or  $\text{GF}_2$  for 150 days in the manner described above. The chambers were pumped down to  $10^{-6}$  torr ( $1.33 \times 10^{-4}$  n/m<sup>2</sup>) and back-filled to  $10^{-3}$  torr ( $.133$  n/m<sup>2</sup>) once a week during the 150 days to take care of leakage. The chambers were purged with  $\text{GN}_2$  at the conclusion of the test.

Environments 8g and 8h. Specimens were suspended in the chambers on racks, the chamber temperature was raised to  $95 \pm 4^\circ\text{F}$  ( $35 \pm 2^\circ\text{C}$ ) using the heater tapes and the chambers were pumped down to  $10^{-6}$  torr ( $1.33 \times 10^{-4}$  n/m<sup>2</sup>). Air was bubbled through a container of water, 18, at  $93^\circ\text{F}$  ( $34^\circ\text{C}$ ) producing saturated air. When the air was introduced into the test chambers at  $95^\circ\text{F}$  ( $35^\circ\text{C}$ ) and one atmosphere pressure, the relative humidity dropped to 95%.

For environment 8g, the moist air is continuously passed through the test chambers. The mix tank is evacuated to  $10^{-6}$  torr ( $1.33 \times 10^{-4}$  n/m<sup>2</sup>) and then back-filled to  $10^{-2}$  torr ( $1.33$  n/m<sup>2</sup>) with  $\text{GF}_2$ . The mix tank pressure is then raised to 30 psia. The  $\text{GN}_2$  and  $\text{GF}_2$  supplies are shut off and the  $\text{GF}_2/\text{GN}_2$  mixture is trickled into the moist air stream at a rate equivalent to  $10^{-3}$  torr ( $.133$  n/m<sup>2</sup>) partial pressure of  $\text{GF}_2$  in the moist air stream. (The  $\text{GF}_2$

in actuality reacts with the moisture forming hydrofluoric acid.) The  $\text{GN}_2$  dilution of  $\text{GF}_2$  is required in order to use practical orifice sizes for flow rate control. The  $\text{GN}_2$  flow rate is small compared to the moist air flow ; consequently, the desired test conditions are not significantly changed by this  $\text{GN}_2$  dilution procedure. The  $\text{GF}_2$ /air flow mixture is continued for four hours.

For environment 8h<sup>(1)</sup>, once the moist air test conditions of 95% relative humidity at 95°F (35°C) are established in the test chambers, the air flow is shut off and  $\text{GF}_2$  is trickled into the test chambers at  $\sim 10 \text{ in}^3/\text{min}$  ( $1.6 \times 10^{-4} \text{ m}^3/\text{min}$ ). This flow rate will essentially just displace the moist air at the end of the four-hour test period, increasing the fluorine partial pressure from 0 to 760 torr ( $10^5 \text{ n/m}^2$ ).

Both of these tests are designed to simulate the effects of different fluorine leak rates into an insulation system on a warm, humid day, assuming the insulation is not protected with an inert gas layer.

---

(1) This exposure was performed only on materials surviving environment 8b.

## Section 4

### PRE-EXPOSURE, IN-SITU AND POST EXPOSURE TESTS

Details on the tests performed before, during and after the environmental exposures are given in this section. The types of tests and number of data points to be obtained for each material was shown previously in Tables 1 through 4.

The tests to be performed are:

- ° Weight and Density
- ° Emittance, Absorptance, Reflectance, Flexibility and Adhesion
- ° Tensile Ultimate
- ° Shear Ultimate
- ° T-Peel
- ° Compression
- ° Ignition
- ° Outgassing

Details of the test specimens used were given previously in Section 2. Tests to be performed on these specimens are given here.

#### 4.1 WEIGHT, W, AND DENSITY, $\rho$ , TESTS

Pre- and post-exposure weight of the specimens are determined using an analytical balance. Dimensions are determined using vernier caliper measurements of width, length, and thickness. Density values are calculated from the weight and dimension measurements.

The accuracy of the weight determination is  $\pm 0.5$  milligrams up to 160 grams.

The accuracy of the dimension measurements was shown previously in Fig. 5.

#### 4.2 NEAR NORMAL EMITTANCE, $\epsilon$

Near-normal reflectance measurements are made on the radiation shield materials and thermal control paints using the Gier-Dunkle Model DB 100 infrared reflectometer shown in Fig. 13. The measurement is independent of sample temperature, and insulating materials and thin films can be measured with no loss of precision.

The reflectometer consists of two cavities maintained at different temperatures. These cavities rotate, and the sample is alternately irradiated by energy from each source temperature. A total detector views the sample and receives energy emitted by the sample and energy reflected by the sample from each cavity (source). Only reflected energy varies as the sample is alternately illuminated by the hot and cold sources; thus, sample temperature is not a consideration.

The accuracy of the device is  $\pm 0.01$  for gray samples and  $\pm 0.03$  for non-gray materials. The measured reflectance is subtracted from 1 to yield the near normal emittance.

#### 4.3 SOLAR REFLECTANCE, $\rho_s$ , AND SOLAR ABSORPTANCE, $\alpha_s$

Direct measurement of solar absorptance is only possible by flight testing (i.e., exposing materials during an actual space flight to the extraterrestrial sun). Because of the enormous cost and complexity of such measurements, a number of ground laboratory techniques have been developed to provide data from which solar absorptance can be inferred. The most commonly used method is to measure the spectral reflectance of the material. For opaque materials, or composites having an opaque rear surface, the spectral absorptance is equal to unity minus the spectral reflectance. (For non-opaque materials such as the radiation shields, only spectral reflectance is reported.) Using data on the spectral irradiance of the extraterrestrial sun (Ref. 2), the spectral

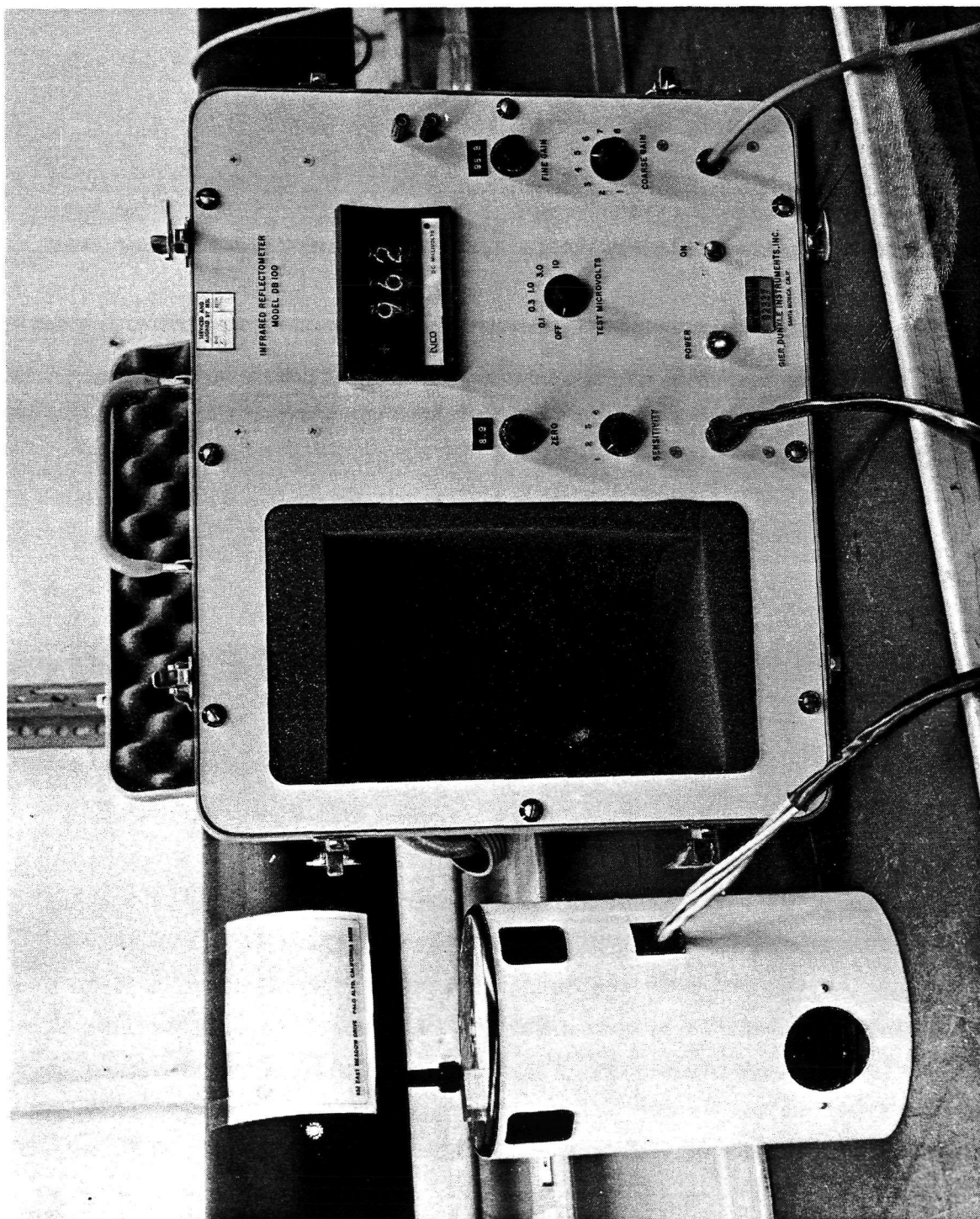


Fig. 13 Gier-Dunkle Model DB 100 Infrared Reflectometer

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absorptance data times the solar spectral irradiance values are integrated over the solar wavelength region (Ref. 3) to yield a solar absorptance.

As the intent of the program was to evaluate changes in properties due to various environmental exposures, the technique chosen for a particular measurement should have good accuracy in detecting changes (i.e., precision), but not necessarily high accuracy in absolute value of the properties, such as solar absorptance. Therefore, a spectral reflectance technique has been chosen for this program to measure pre- and post-test values, which is highly reproducible ( $\alpha_s$  to 0.01) and of reasonably good accuracy ( $\alpha_s$  to  $\pm 0.03$  for  $\alpha_s = 0.15$  to 1.0).

The Cary Model 14 spectrophotometer with the #1411 reflectance attachment shown in Fig. 14, permits relatively rapid determination of changes in spectral reflectance. The specimen is placed over a port in the side of the sphere and illuminated by the external monochromatic source. Energy reflected from the specimen is then reflected on to the detector (0.18 to 2.7 microns). The resulting spectral data are then integrated against the Johnson curve (Ref. 1) to obtain solar reflectance (radiation shields), and solar absorptance (thermal control paints) is inferred from Kirchhoff's law. The accuracy of the measurement is  $\pm 0.02$  absorptance units.

#### 4.4 FLEXIBILITY, $F_b$

The flexibility test for the radiation shield and Teflon film materials consists of folding a specimen 180 degrees and applying light pressure to the fold area with a two-inch diameter 4.5 lb roller. The specimen is then unfolded and examined visually for evidence of substrate or film cracking and coating separation (where applicable).



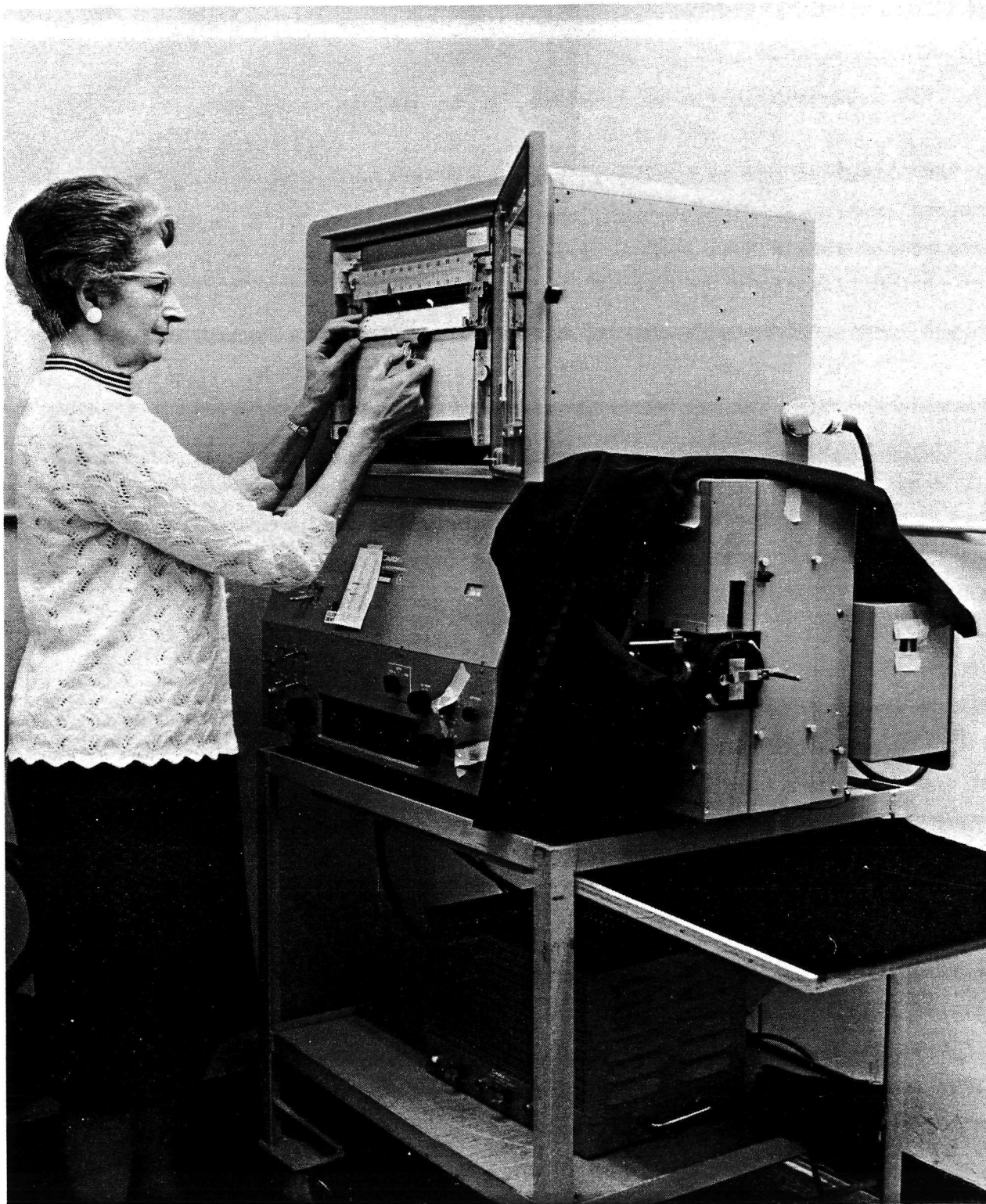


Fig. 14 Cary Model 14 Spectrophotometer With the #1411 Reflectance Attachment

#### 4.5 ADHESION, AD

Adhesion tests are performed on the metallized radiation shields and the thermal control paints. Adhesion tests are performed no closer than one inch from any edge of the test specimen. Coating adhesion is verified by the procedure outlined below:

- ° Adhesive tape, Minnesota Mining & Manufacturing Company Code No. 250, or equivalent, 1-inch wide and at least 2-inches long, is applied, adhesive side down, to the coated surface and rolled twice with a two-inch diameter 4.5 pound roller.
- ° The tape is removed with a single, abrupt movement perpendicular to the surface.
- ° The tape surface is then examined visually for coating separation or damage and the percentage of coating removed is estimated and recorded.

#### 4.6 TENSILE ULTIMATE, $F_t$

The following procedure applies to the radiation shields, TFE film, nettings and Beta Glass cloth. Exceptions to the procedure for the Tissuglas spacer are described where appropriate. Specimen grips were prepared for receiving the specimen as follows:

- ° A "side bar" was used to hold the two grips correctly aligned and the correct distance apart. This side bar was fastened to the grips by two socket-head screws which were only finger tight (for easy unfastening).



- ° Shims of the proper height were placed between the two grips such that when the two grips and these shims are placed on a flat working surface, they present a smooth and stepless surface across the span covered by the specimen.

The specimen was aligned on the grips and the two clamping plates installed. (The Tissuglas was attached to the grips with adhesive tape.) Care was used to avoid any significant pretensioning of the specimen, although a very gentle application of tension was necessary to avoid any ripples in the specimen. The presence of any ripples in the clamped specimen was eliminated by removing the clamp and reclamping after additional tension had been applied to remove the ripple. While it is impossible to prescribe (or measure) any initial "pre-tension" in the specimen, it was found that an experienced technician can develop a "feel" for the amount of pull required. It is estimated that this pull is less than 0.1 lb (.4 n).

The grips containing the specimen (and still held apart the correct distance by the side bar) were installed in the tensile tester as shown in Fig. 15. The position of the movable attachment clevis on the tester was such that the clevis pin was inserted without force.

After both clevis pins were installed, the side bar was removed. Care was taken so as not to move either grip in any direction during the removal of the side bars.

The movable clevis pin was made to move in a direction opposite to that required to apply tension to the specimen. This in effect causes the specimen to become slack and removes from the specimen any tension that may be caused by the weight of the lower grip. The removal of all tension in the specimen was marked by no further decrease in load readout. The pen on the load recorder was prepositioned to allow for the movement of the pen in a decreasing load direction. The cross-head (or clevis) motion was then reversed (i.e.,

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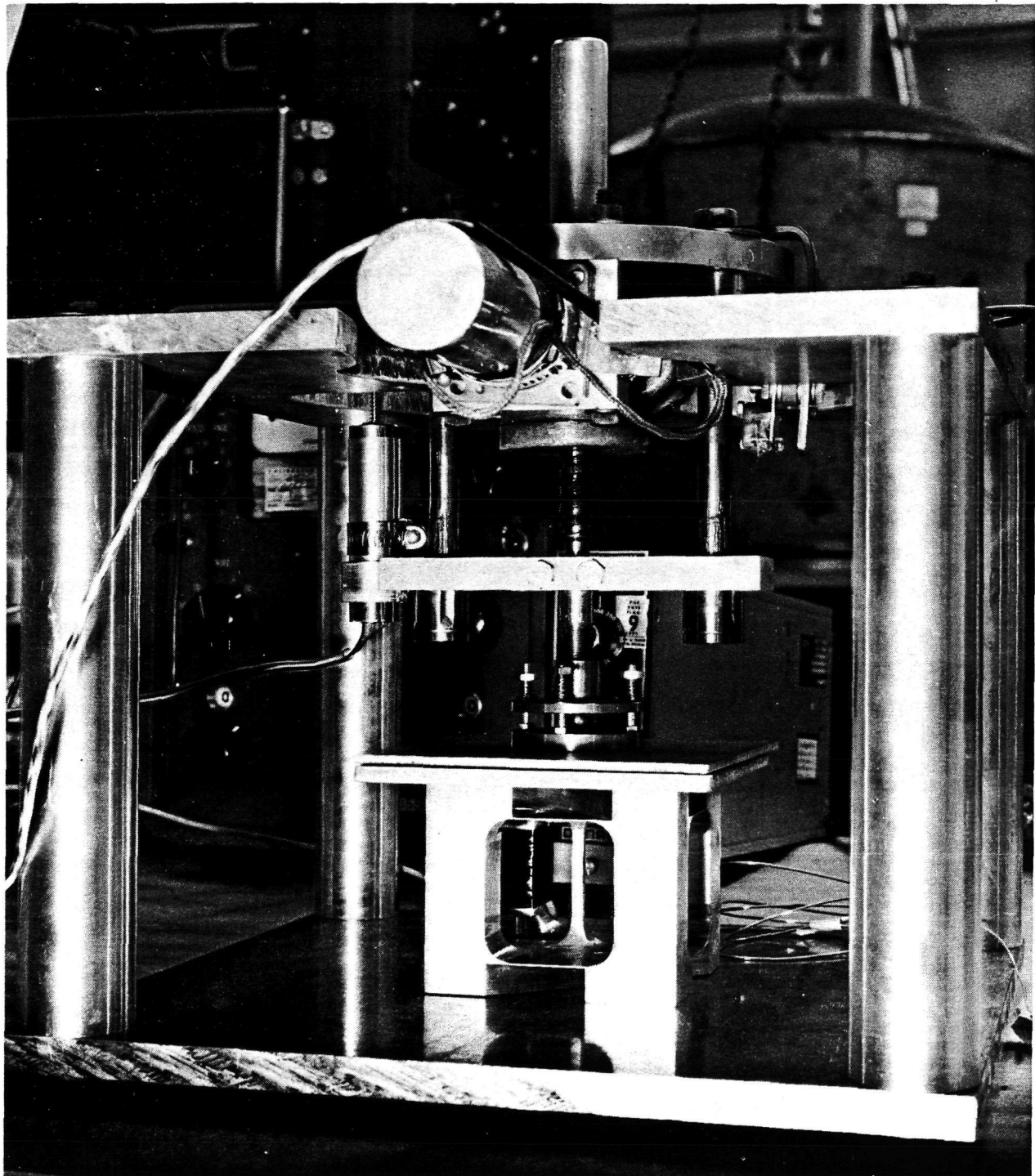


Fig. 15 Tensile Test Apparatus

made to move in a direction which induced tension). When the slack in the specimen was removed, the load started to increase. For some materials this onset of load was very gradual - notably the mesh materials.

Clevis movement normally stopped just as soon as loading of the specimen started. The application of tension was backed off somewhat if any measurable load was apparent. The load recording instrument was then "zeroed" and calibrated. (A dynamometer was used in place of the load cell for the Tissuglas specimens.)

The load cell system was calibrated by shunting of the load cell bridge. For load cells below 200 lb (890 n), the load cell was calibrated with dead weights, and a shunt resistor across the appropriate bridge leg was found which produces the same output signal as the calibration load. The weights are accurate to 0.25%, and a digital voltmeter was used to read the load cell signal which was then read to 0.1% accuracy. (The dynamometer is accurate to  $\pm 1\%$  of full scale.) The same resistor (or same setting on the same precision resistance box) was then used to shunt the load cell bridge once it was connected to the load indicator used in the test (an X-Y plotter).

The specimen was then loaded to failure at a rate which caused failure after 20 to 60 seconds of loading. In this range, the rate of loading is not critical, but was kept uniform. Uniformity of loading rate was achieved by maintaining a constant voltage to the D.C. drive motor of the testing machine. This motor is geared down so that the loading process represents no significant load on the motor.

After failure, the specimen was removed from the grips and inspected for any possible abnormalities in the failure, which were reported if they occurred. Maximum load was noted on the records.

Accuracy of the failure load is 0.5 percent of the maximum value of the range used ( $\pm 1\%$  for the Tissuglas specimens).

#### 4.7 SHEAR ULTIMATE, $F_s$

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The procedure for pulling the adhesive and Velcro shear strips was generally similar to that used in the standard tension tests described previously. The following items are exceptions and special precautions were observed:

- ° The length of the overlap zone was measured.
- ° No side bar was required on the specimen.
- ° For the adhesive specimens, the load measuring system zero was obtained by removing the clevis pin.
- ° The accuracies given formerly still apply.

#### 4.8 T-PEEL, $F_p$

The following test procedure was used: Each half of the Velcro fastener was bonded to its attachment plate as shown in Fig. 16. The attachment plate is made from 0.50-in. (1.27-cm) aluminum. The attachment plates were connected to their respective connector bracket, and the assembly was placed in the testing machine. The testing machine cross-head was adjusted so that the clevis pin slipped easily through the connector brackets. The load measuring system was zeroed and calibrated as required. (Zero load setting was made with one of the clevis pins removed.) The tensile force was applied across the fastener. The loading rate was uniform such that failure occurred in 20 to 60 seconds after load application starts. Accuracy of the measurement was  $\pm 0.5$  percent of full scale.

#### 4.9 COMPRESSION TESTS, LD AND $F_c$

Compression tests, required on the spacer materials and the rigid, closed-cell, polyurethane foam are somewhat different and are described separately.

Spacer Materials, LD. These tests were performed on the Instron machine using 20 test layers. The layers were sandwiched between two rigid steel



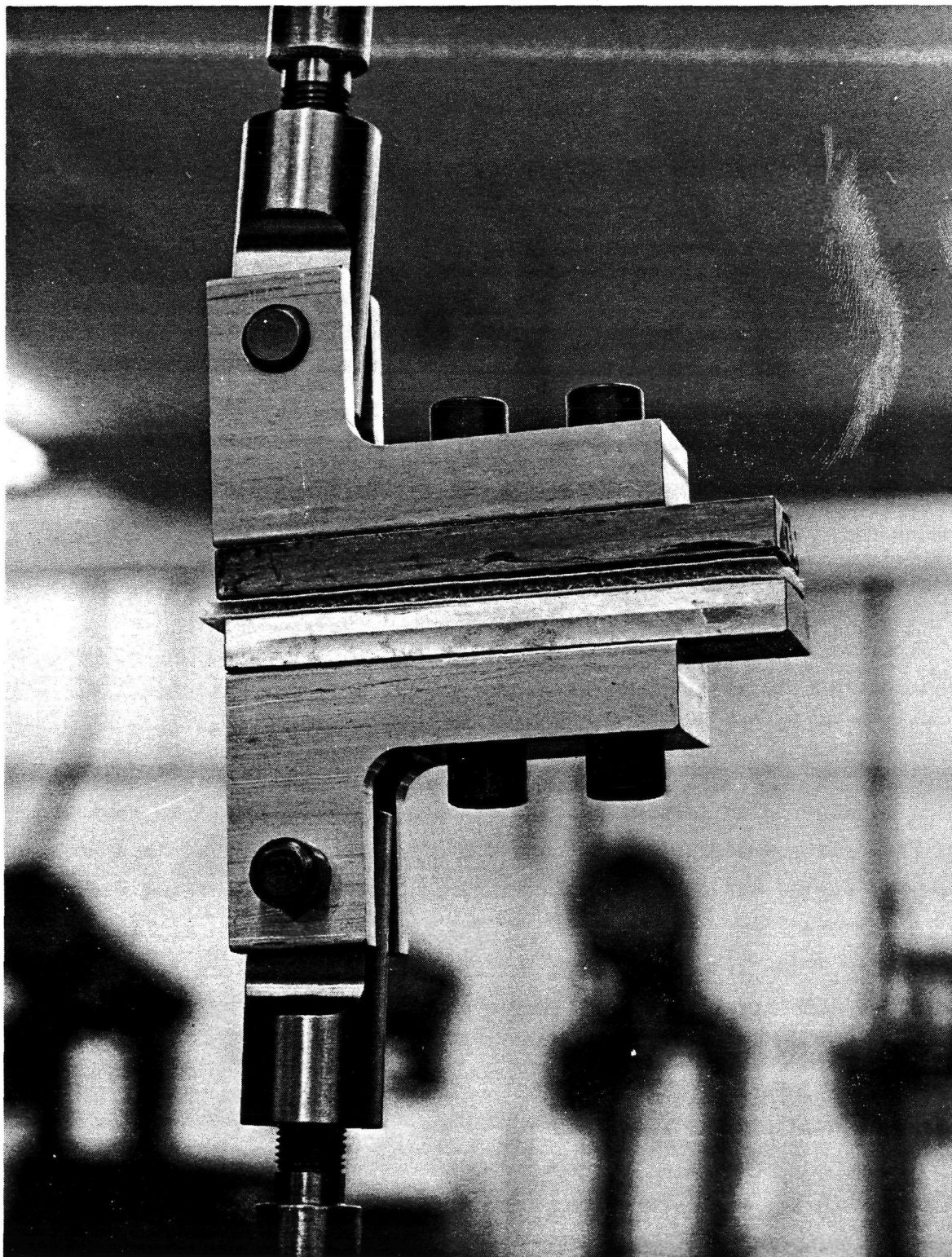


Fig. 16 T-Peel Test Fixture

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plates as shown in Fig. 17 and the free standing uncompressed sample height was measured with the top steel plate touching the top spacer layer at a minimum of three points. A compression load was then applied to the spacers until 50% deflection was reached. The movement was recorded on an X-Y plotter. The load was held for one minute and released. The free standing height (T) was then measured as described previously to  $\pm 0.001$ -in. ( $\pm 0.0025$ -cm). The layer density was calculated by dividing T by the number of layers tested (twenty).

Polyurethane Foam,  $F_c$ . These tests also used the Instron machine. The specimen was placed between the steel plates with the .625 in. (1.59 cm) dimension perpendicular to the plates. The load versus deflection curve was plotted. Failure was noted where a rapid change in slope occurred in the load-deflection curve.

#### 4.10 IGNITION, IG

The ignition tests were discussed as a part of the environmental propellant exposure in Section 3.8.

#### 4.11 OUTGASSING, OG

The outgassing data were obtained for Environments 2b 660°R (366°K), 2c 530°R (294°K), 2d 140°R (78°K) and 4 (nitrogen and helium preconditioning). First, the experimental approach, test apparatus and instrumentation required to perform the experiment are discussed followed by the test procedure used.

##### 4.11.1 Experimental Approach

The basic technique used to determine outgassing rates was the throughput method, in which a vacuum chamber containing an insulation sample is evacuated and the pressure differential across the evacuation line is measured.

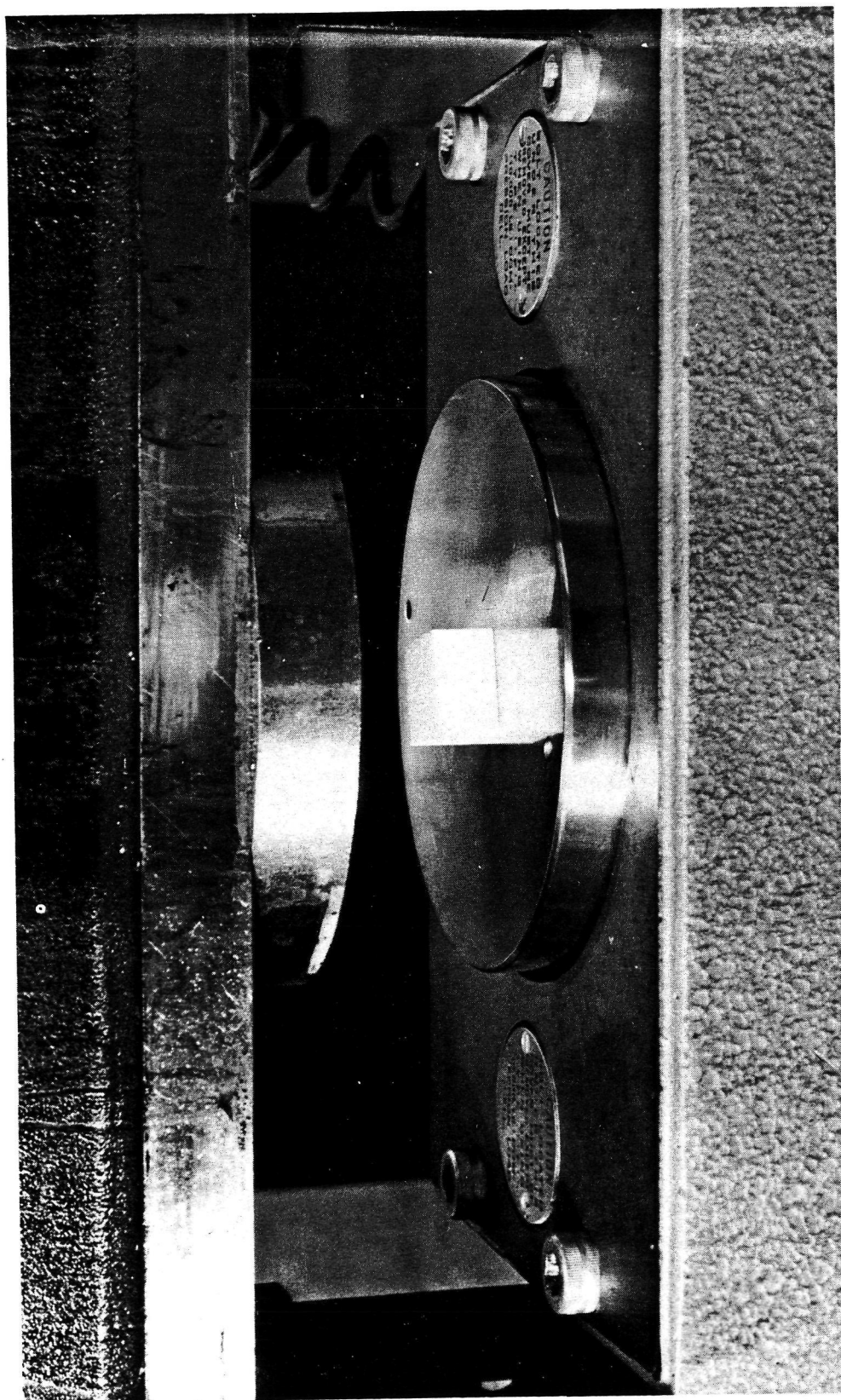


Fig. 17 Compression Test Setup for Spacer or Foam Materials

Since the flow conductance of the evacuation line was determined independently, the gas flow rate from the chamber could be determined from the known conductance and the measured pressure differential. The gas flow in the evacuation line consists of sample outgassing plus the background outgassing from the vacuum chamber and its instrumentation. Hence, it was necessary to perform tests with the vacuum chamber empty to determine the magnitude of the background outgassing rate in order to subtract it from the measured total. The flow conductance of the evacuation line is a function of the molecular weight of the gas so the vacuum chambers were equipped with the instrumentation necessary to determine the gas species present.

#### 4.11.2 Throughput Method Theory

The throughput method requires that separate evacuation experiments be run for the empty vacuum chamber and the chamber plus sample. The mass balance equations for vacuum chamber under these two conditions are:

$$-\frac{V_c}{RT_c} \left( \frac{dP_{co}}{dt} \right) = S \frac{(P_{co} - P_{do})}{RT_p} - Q_c \quad (\text{Empty Chamber}) \quad (4-1)$$

$$-\frac{V_c}{RT_c} \left( \frac{dP_{cs}}{dt} \right) = S \frac{(P_{cs} - P_{ds})}{RT_p} - Q_c - Q_s \quad (\text{Chamber Plus Sample}) \quad (4-2)$$

where

$V_c$	=	sample chamber volume
$T_c$	=	sample temperature
$T_p$	=	pumping line temperature
$S$	=	volumetric pumping speed, volume per unit time
$Q_c, Q_s$	=	chamber, sample outgassing rates, mass released per unit time
$P_{co}$	=	empty chamber gas pressure
$P_{cs}$	=	chamber pressure with sample in place
$P_{do}$	=	downstream gas pressure for empty chamber
$P_{ds}$	=	downstream gas pressure with sample in place
$t$	=	time
$R$	=	gas constant for species outgassed



The chamber outgassing rate,  $Q_c$ , is determined from the empty chamber experiment and equation 4-1. After times of one hour or more the pressure transient terms on the lefthand side of equations 4-1 and 4-2 are negligible.

$Q_c$  is then given by:

$$Q_c = S/RT_p [P_{co} - P_{do}] \quad (4-3)$$

Inserting this value in equation 4-2 and noting that all pressures relate to the same time since beginning of evacuation, one obtains the following expression for sample gross outgassing rate

$$Q_s(t) = \frac{S}{RT_p} \left\{ P_{cs}(t) - P_{ds}(t) - [P_{co}(t) - P_{do}(t)] \right\} \quad (4-4)$$

The partial pressures of each gas species attributable to the sample alone,  $P_i$ , are found by subtracting the partial pressure of each component for the empty chamber,  $P_{i0}$ , from the partial pressure of the same component for the case of chamber plus sample,  $P_{is}$ . The fraction of the  $i$ th component,  $F_i$ , is found from the following expression

$$F_i = \frac{P_i M_i}{\sum_{j=1}^n P_j M_j} \quad (4-5)$$

The average molecular weight of the gas being pumped,  $\bar{M}$ , is found from the following expression

$$\bar{M} = \frac{\sum_{j=1}^n P_j M_j}{\sum_{j=1}^n P_j} \quad (4-6)$$

#### 4.11.3 Test Apparatus

The test apparatus is shown in Fig. 18. The apparatus consists basically of four identical vacuum test chambers shown in Fig. 19 connected radially to a central diffusion pumping system. Four chambers were used in order to compress the test schedule. They can be opened and closed for sample insertion and removal by means of a flanged joint. The flange is sealed with a Viton O-ring for the 660°R (366°K) and 530°R (294°K) tests, and with a copper gasket for the 140°R (78°K) tests.

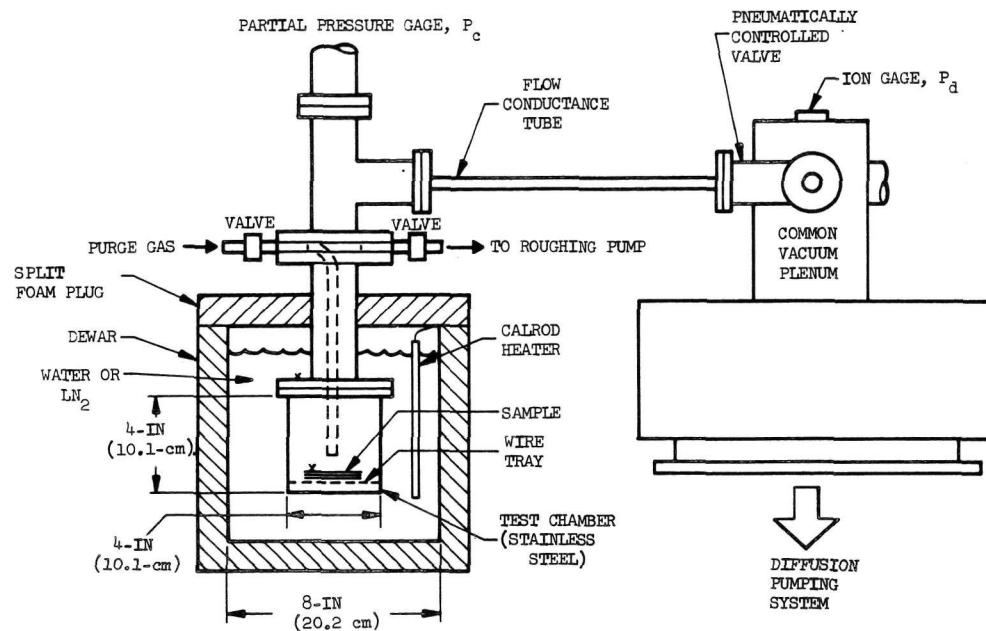
The test chamber is evacuated via an evacuation flow conductance tube whose conductance was selected so as to maintain the vacuum chamber pressure in the range of  $10^{-5}$  torr to  $10^{-7}$  torr ( $1.3 \times 10^{-3}$  to  $1.3 \times 10^{-5}$  n/m<sup>2</sup>) for the greater part of the tests.

A solenoid-controlled pneumatically-operated valve is included in the evacuation line at its downstream end. This valve can be used to isolate the individual chambers so that they can be pressurized for sample turnaround. Also, the valve is designed to close in the event of a power failure so as to protect the experiment.

The same chamber pressure is measured with a Varian partial pressure gage<sup>1</sup> which is attached to the vacuum chamber system via a tee at the sample chamber/flow conductance tube interface. This gage is a modification of the standard ion gage which can read partial pressure of individual species as well as total pressure. The gage is a type of mass spectrometer with two

---

1 At the beginning of the environment 4b tests frequent failure of the tungsten filaments occurred. (Environment 2b, 2c and 2d tests were already complete.) The 4b tests were eventually completed after many delays and filament failures. Between the 4b and 4a tests the gages were fitted with thoriated-iridium filaments. All 4a tests were conducted with the iridium filaments and no further filament failures occurred. The technical background to this problem is presented in Ref 5.



NOTE: LOCATION OF  $Cu/Cn$  THERMOCOUPLES ON ONE CHAMBER (PRIOR TO TESTS)  
ONLY TOP THERMOCOUPLE REMAINS FOR OUTGASSING TESTS

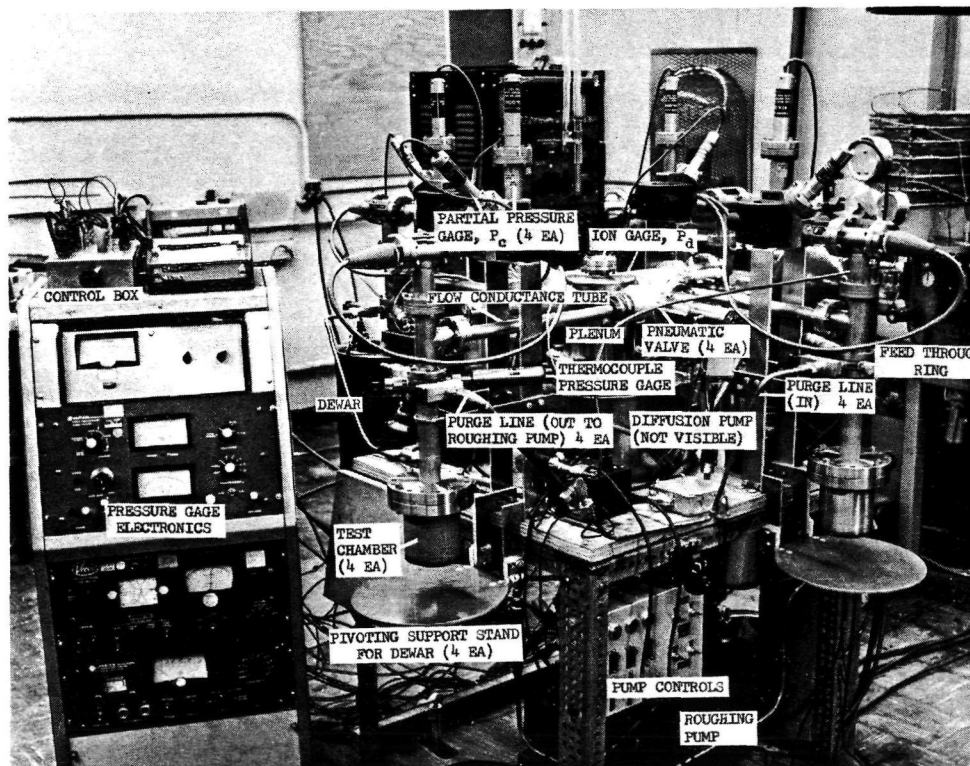


Fig. 18 Outgassing Apparatus

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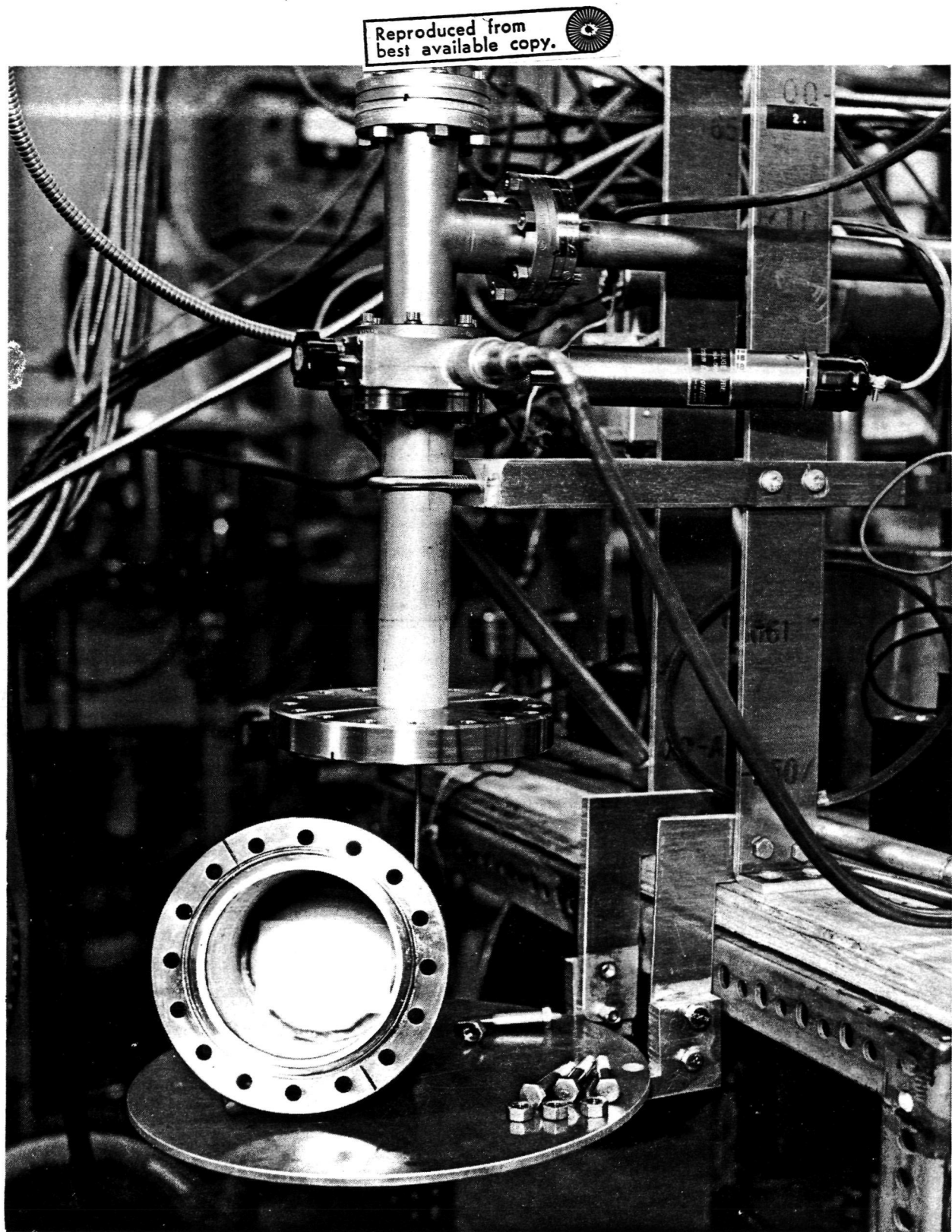


Fig. 19 Test Specimen in Outgassing Chamber

ion collectors arranged so that it operates as a 135° instrument for masses 1-10 amu and as a 90° instrument for masses 10-70 amu. The gage has an ion source which also serves as the total pressure gage. The ionized gas molecules are focused into a beam and are directed towards the magnetic analyzer. Mass scanning is achieved by varying the accelerating voltage. The resolution capability of the partial pressure gage is best at lower mass numbers but is not satisfactory at mass numbers much greater than about 50 amu. In the present tests the principal gas components were water vapor (amu = 18), nitrogen (amu = 14), and carbon dioxide (amu = 44), which were well within the capacity of the gage. The four partial pressure gage heads were controlled from a single control box with the use of a four position selector switch.

The pressure downstream of the flow conductance evacuation tube was measured in the diffusion pump plenum with an ion gage. Additional services to the sample vacuum chamber were provided via a feedthrough ring at the chamber/partial pressure gage/evacuation line tee. A Bendix Magnevac thermocouple gage served to monitor the chamber pressure at pressures between one atmosphere and about  $10^{-4}$  torr ( $.0133 \text{ n/m}^2$ ). Purge gas could be fed to the chamber through a line in the feedthrough, and could be removed via a second line which also served as a roughing line when the chamber was to be evacuated. A thermocouple temperature probe feedthrough was also provided.

For tests at 660°R (366°K) the temperature of the vacuum chambers was raised by immersing them in water-filled temperature-controlled baths. A layer of oil over the water minimized evaporation of the water. For tests at 140°R (78°K) the chambers were cooled in liquid nitrogen filled, foam insulated, stainless steel dewars.

#### 4.11.4 Test Procedure

Certain preliminary tests and observations were made before beginning the insulation test program. After initial functional checkout of the apparatus, it was found that the downstream pressure,  $P_d$ , was invariably less than 1 percent of the chamber pressure,  $P_c$ . Hence,  $P_d$  was considered to be negligible and was not measured during the insulation test program.

The pumping speed of the sample chamber evacuation line,  $S$ , was then determined both by calculation and by a close-off experiment. The equation used to calculate the pumping speed,  $S$ , was taken from Dushman (Ref 4, Eq. 2.38).

$$S = 364 \left( \frac{1}{1 + 3/8 \cdot \ell/a} \right) (\pi a^2) \left( \frac{T}{M} \right)^{1/2} \quad (4-7)$$

The tube length and radius are  $\ell$  and  $a$ , respectively. The temperature is  $T$  and the molecular weight is  $M$ . The speed was determined separately for each homogeneous section of the line and were added together reciprocally  $\frac{1}{S_1} + \frac{1}{S_2} + \dots + \frac{1}{S_n}$ . The result was then checked by isolating a chamber from the pumping system and observing its rate of pressure rise. The following mass balance equation can be written if it is assumed that the gas generation rate is the same just before and just after closure. The relationship equates the quasi-steady state flow rate before close-off to the rate of gas accumulation just after close off.

$$(P_{co} - P_{do}) S = V_c \left( \frac{dP_{co}}{dt} \right) \quad (4-8)$$

$V_c$  is the chamber volume,  $P_{co}$  is the empty chamber pressure,  $P_{do}$  is the pressure downstream of the flow conductance  $S$ , and  $t$  is time. The experimental measurements confirmed the calculation to within 15 percent. The pumping speed determined from these calculations and measurements is  $3.1 \pm .25$   $\ell/\text{sec}$  for a molecular weight of 18 (water vapor).



In certain tests it was necessary to raise or lower the sample chamber temperature before beginning pump down. However, continuous temperature measurement within the test chamber by the thermocouples, for example, would have meant introducing an additional source of background outgassing as well as requiring the taking of essentially unimportant data. Instead, preliminary tests were conducted in which one chamber was fitted with a set of thermocouples to measure chamber bottom, chamber top and sample temperatures. The chamber was evacuated and then heated or cooled and the time required for all three thermocouples to show temperature equilibrium (with the hot water or liquid nitrogen bath) was recorded. In both cases these times were less than one-half hour. The bath was then removed and the times needed to re-attain ambient conditions were measured. These times were three-four hours. The thermocouples were removed and the temperature transient time periods were included in the test program. In the actual test program the sample chamber was filled with helium during these transients so the transient periods would be shorter than the previously measured values.

After these preliminary experiments the test program proper was begun. Empty chamber tests were made for each chamber at each environment before and after the completed sample test program in that environment. The step by step procedure followed for each test was as follows:

The size of each insulation material sample was chosen so as to provide a total outgassing rate hopefully not too low to be measured and not too high to keep the chamber pressure above a measurable value. Since such an estimate must be based upon a knowledge of the outgassing rate, as yet unknown, the sample size was based upon published data for similar materials. The sample weight and nominal exposed surface area were measured. The surface area of samples such as adhesives, paints and polyurethane foam are obviously not characteristic dimensions and were recorded only for completeness. All sample handling was done with clean nylon gloves and/or clean tongs.

The sample chamber was brought to ambient temperature, when necessary, and

its pressure was raised to one atmosphere by admitting helium (nitrogen for environment 4a). The lower section of the chamber was unbolted and lowered a distance of about 2-in. (5.1-cm). A flow of helium (nitrogen for 4a) was maintained through the purge line during this operation. The sample already in the chamber was removed and the new sample was inserted. The chamber was then reclosed and the removed sample was weighed.

The sample chamber was then subjected to the environment of interest before the test evacuation. The following procedures were adopted for each environment:

Environment 2b. The insulated bath was placed around the chamber and the water bath temperature raised to 660°R (366°K) by activating the electric heaters. The chamber pressure was maintained at one atmosphere during heating. After one hour of heating the chamber and its contents were assumed to be at 660°R (366°K) and test pumping was begun.

Environment 2c. Test pumping was begun immediately after closure of the chamber.

Environment 2d. The insulated bath was placed around the chamber and filled with liquid nitrogen while the chamber pressure was maintained at one atmosphere of helium. After one hour the chamber and its contents were assumed to be at 140°R (78°K) and test pumping was begun.

Environment 4a. The warm-up procedure described for environment 2b was followed. Pumping was then initiated and maintained for six hours. No data were recorded during this period. At the end of six hours, the chamber was isolated from the vacuum pumping system, the hot bath was removed, and the chamber allowed to cool down overnight. The following morning, thirteen hours later, dry nitrogen



at one atmosphere pressure was flowed slowly through the sample chamber for four hours. At the end of this period test pumping was begun.

Environment 4b. The procedure was identical to that for environment 4a, with the single exception that helium was used for the four hour purge process.

At the beginning of test pumping, the time was recorded and the roughing valve to the chamber of interest was opened. When the chamber pressure has fallen to below  $5 \times 10^{-2}$  torr ( $6.7 \text{ n/m}^2$ ) the downstream pressure was noted and the pneumatic valves to the three other test chambers were closed. The roughing valve was closed and the pneumatic valve was opened. When the downstream pressure had fallen to approximately the value it had just before the valve switching process, the pneumatic valves to the other three chambers were reopened. This procedure took two to three minutes and was adopted to prevent contamination of previous test specimens by the new specimen.

When the chamber pressure had fallen to a value below  $10^{-4}$  torr ( $.013 \text{ n/m}^2$ ) the partial pressure gage was turned on. Data were taken at one-half to one hour intervals for the remainder of one working day, usually about six hours. Data points were also taken on the 2nd, 4th and 7th days of the tests. One data point consisted of the time, the total pressure and the partial pressures of the gas components. It was found that the only significant gas components were water vapor, nitrogen and carbon dioxide so in practice, data gathering was confined to these three gases.

At the end of the 7-day test period the final data points were taken. The chamber was isolated from the main pumping system. For environment 2b the constant temperature bath was removed and the chamber was returned to ambient temperature. The chamber pressure was raised to one atmosphere with helium, the chamber was opened and the sample weight was determined.

#### 4.11.5 Data Reduction

The outgassing rates and gas fractions present for the insulation samples were calculated from the above listed experimental data as follows: The empty chamber pressure history data were curve-fitted using a digital computer to give expressions of the form

$$\log P = a_0 + a_1 \log t + a_2 (\log t)^2 + a_3 (\log t)^3 \quad (4-9)$$

where  $a_0$ ,  $a_1$ ,  $a_2$  and  $a_3$  are constants. Expressions of this type were generated for total pressure and three partial pressure histories for each chamber and each environment. The coefficients for the empty chamber data curve-fits were built into OUTGAS, a digital computer program for reducing the insulation sample data. The insulation sample pressures and times were read as input to OUTGAS which calculated outgassing rate and water vapor, nitrogen and carbon dioxide fractions for each data time point. The calculation steps are as follows:

- ° Determine total and partial pressure contribution of the insulation sample at each experiment point in time by subtracting the empty chamber pressure, calculated from the appropriate curve fit for this point in time, from the measured pressure data.
- ° Using the corrected partial pressure data, calculate mass fractions of each of these three measured gas species and the average molecular weight.
- ° Using the calculated average molecular weight, the evacuation tube flow conductances were corrected and the gas constant R determined.
- ° Using equation 4-5 with  $T_p$  equal to 529°R (294°K) the outgassing rate  $Q_g(t)$  was determined.

- ° The specific outgassing rate in gms/sec per unit area or per unit weight was calculated by dividing the  $Q_g(t)$  by the given area or given weight. In general, all data were expressed on an area basis except where this method made little sense. The data for the following materials were calculated on a weight basis: NA, GY, PF, V, TH-A and BL-A.

#### 4.11.6 Data Accuracy

Assuming the general validity of the throughput method and equation 4-5, the absolute accuracy of the outgassing rate versus time is a function of the pressure measurements, evacuation tube conductance, average molecular weight of the desorbed gas, sample area and weight, and time. The time, sample weight and area measurements can be assumed to be of better than 1 percent absolute accuracy and are not factors in determining the overall accuracy. The calculation of the average molecular weight is based upon partial pressure measurements, so the overall accuracy depends only upon the pressure measurements and the evacuation tube conductance. The latter was calculated and verified experimentally to within  $\pm 8$  percent. The ion gage was periodically calibrated against a McLeod gage which brings the error to a few percent or less. The absolute accuracy of the partial pressure measurements is immaterial since their only purpose is to permit gas component fractions to be determined. It may be concluded therefore that the theoretical accuracy of the pressure measurements and the gas component fractions is of the order of a few percent.

An additional +5% error is estimated due to possible non-steady state conditions (inadequate time for filament warm-up). The size of the error is estimated by observing the deviation of data points from expected smooth curves, and from general experience with the apparatus.

## Section 5

### SUMMARY OF TEST RESULTS

Due to the magnitude of the test data obtained on this program, the presentation of the tabular and graphical test results are provided in a separate Volume (No. II). A summary of the test results are presented in this section.

A criterion used to determine if a significant property change has occurred (due to the environmental exposure) is given first followed by the calculated control sample error bands used as this judgment criteria for all test materials. Next, the post-exposure, test-material, property-values that fall outside the control sample error bands are presented as a percent change from the pre-exposure or control value for all properties measured (except oxygen and fluorine ignition tests and outgassing tests where percent changes are not applicable). The ignition and outgassing test results are discussed separately.

#### 5.1 ESTABLISHMENT OF CONTROL SAMPLE STANDARDS (ENVIRONMENT 1)

In order to determine whether property changes noted in post-exposure test samples are due to the effects of environmental exposure or just the normal statistical variation about a mean, it is necessary to compare these values against a standard. This standard is obtained from the 0, 10 and 150-day control sample data measured for all the test materials. For each control specimen property, a mean value and a standard error about that mean can be calculated. Post-exposure test values falling outside this error band are considered to have undergone a significant property change due to the effects of the environmental exposure.

In calculating the standard error band, three items must be considered:

1. The deviation from the normal distribution due to the small

sample sizes used in the tests (ranging from 1 to 6 data points per property).

2. The possible shift in the control value property with time due to aging over the 150-day time span.
3. Use of the most suitable probability of occurrence (in terms of number of standard errors) that will encompass only the normal statistical variations about a mean and will not mask out actual test trends.

To correct for the small sample size, a  $t/2$  distribution correction factor is used as shown below:

$$\sigma = \frac{t}{2} \sqrt{\frac{\sum(X - \bar{X})^2}{n-1}}$$

where

- $\sigma$  = standard error
- $t$  =  $t$  distribution (sampling distribution correction for a small sample size, see Table 7)
- $X$  = data point
- $\bar{X}$  = mean value of data
- $n$  = number of data points

Table 7  
SAMPLE SIZE CORRECTION FACTORS

NO. OF DATA POINTS	$t/2$	
	$1\sigma$	$2\sigma$
2	1.41	7.05
3	1.15	2.29
4	1.09	1.64
5	1.06	1.40
6	1.05	1.31

This correction takes into account the increasing uncertainty in the true property value range as the sample size decreases. Sample sizes of ~30 or more are required before this correction factor becomes insignificant.

Inherent in the assumptions used in calculating error bands for combined control data from the 0, 10, and 150 day samples is that no significantly large shifts occur with time in property values to skew the normal distribution. Because there are only a maximum of two data points at each time period (0, 10 and 150 days) separate standard error calculations are not as meaningful as where a larger number of data points are used. For most of the control data, using a combination of the 0, 10 and 150 day results appears to be a valid assumption and the error band appears reasonable narrow. Exceptions to this conclusion are discussed later.

Both one ( $1\sigma$ ) and two ( $2\sigma$ ) standard error bands for the controls were considered as the standard criteria for determining whether a significant change has occurred when comparing the post-exposure test values with the control values. For the control samples, a two  $\sigma$  band was selected. Statistically, a two  $\sigma$  band indicates the odds are 21 to 1 against a post-exposure test value falling outside this band due only to statistical fluctuations.

The calculated standard error bands for the control samples are provided in Tables 8 through 11 for the four classes of materials tested. For the non-destructive tests where property measurements were made both before and after an exposure on the same sample (weight, density, reflectance, absorbance and emittance), a delta value was used (10 or 150 day data minus 0 day data). For the other destructive property tests (tensile, tensile shear, peel, compressive strength and layer density) absolute test values were used for the 0, 10 and 150 day data.

For most of the data, using a combination of the 0, 10 and 150 day results appears to be a valid assumption and the  $2\sigma$  spread appears reasonably narrow.

Apparent exceptions noted to the above assumption occur for the emittance values of the radiation shields and the tensile shear strength of the Goodyear adhesive. For radiation shield emittance values at 0 and 10 days, no apparent difference was noted. However, for the 150 day exposures (1c), the emittance values increased by approximately .01 units for all the radiation shields (aluminum and gold coated). The 1c emittance values were rechecked and found to be accurate according to the reference standard and measuring apparatus used. Since a shift of .01 units is within the accuracy of the combined standard/measuring unit procedure used it is highly unlikely that all 16 samples for both aluminum and gold coatings would increase the same amount (or at all based on previous test data). It is felt the increase is due to a shift in the instrument calibration. Since this shift would also have occurred for all the environmental samples measured as well, it is felt that using the combined values of 1a, 1b and 1c will still produce an emittance band most representative over the time period of concern. Even with this shift, the  $2\sigma$  bands for the radiation shields are still relatively narrow, as shown in Table 8.

For the Goodyear tensile shear coupons, the shear strength apparently increased with age as shown in Fig. 20. Using combined 1a, 1b, and 1c data produces a wide  $2\sigma$  band. In this case, it is plausible the adhesive could have cured with age and the shear strength increased with time. For this case, Fig. 20 is used as a reference. The Goodyear adhesive environmental exposure data was compared with the control values based on the calendar time when it was performed. The  $2\sigma$  error band was assumed to be the same as for the Narmco adhesive ( $\pm 15\%$ ).



Table 8

RADIATION SHIELD CONTROL SAMPLE STANDARD ERROR BANDS (2 $\sigma$ )<sup>1</sup>

M A T E R I A L	P R O P E R T Y			
	$\Delta$ WEIGHT GRAMS	TENSILE ULTIMATE LB/IN (N/M)	$\Delta$ REFLECTANCE	$\Delta$ EMITTANCE
S-A-M (Single Aluminized Mylar)	$-.0004 \pm .0005$	$5.3 \pm 1$ (928 $\pm$ 175)	$-.020 \pm .019$	$+.004 \pm .010$
D-A-M (L) <sup>2</sup> (Double Aluminized Mylar)	$-.00003 \pm .00043$	$3.85 \pm .25$ (674 $\pm$ 44)	-	$+.006 \pm .012$
D-A-M (Double Aluminized Mylar)	-	-	-	$+.007 \pm .015$
S-A-K (Single Aluminized Kapton)	$-.0004 \pm .0005$	$3.69 \pm .68$ (646 $\pm$ 119)	$-.023 \pm .041$	$+.004 \pm .010$
S-G-M (Single Goldized Mylar)	$0 \pm .0013$	$3.97 \pm .37$ (695 $\pm$ 65)	$+.0025 \pm .027$	$+.006 \pm .008$
D-G-M (L) (Double Goldized Mylar)	$0 \pm .001$	$4.15 \pm .62$ (727 $\pm$ 109)	-	$+.003 \pm .008$
D-G-M (Double Goldized Mylar)	-	-	-	$+.004 \pm .009$
S-G-K (Single Goldized Kapton)	$+.0025 \pm .0023$	$4.25 \pm .38$ (744 $\pm$ 67)	$-.006 \pm .018$	$+.008 \pm .010$

1. Probability of occurrence of a deviation greater than the band shown due to random fluctuations is 4.6% (for a 2 $\sigma$  band).
2. Coatings tested on each side separately. L refers to marking pattern on shield.



Table 9  
SPACER CONTROL SAMPLE STANDARD ERROR BANDS<sup>1</sup>

M A T E R I A L	P R O P E R T Y		
	Δ WEIGHT GRAMS	TENSILE ULTIMATE, LB/IN (N/M)	LAYER DENSITY NO/IN (NO/CM)
N N (Nylon Net)	+ .0065 ± .0246	2.61 ± .44 (457 ± 77)	123 ± 9 (48 ± 4)
D N (Dacron Net)	- .0098 ± .0180	4.21 ± .52 (737 ± 91)	171 ± 35 (67 ± 14)
S N (Silk Net)	- .0028 ± .0130	2.08 ± .20 (364 ± 35)	214 ± 61 (84 ± 24)
T G (Tissuglas)	- .00195 ± .0005	.27 ± .18 (47 ± 32)	285 ± 36 (112 ± 14)

1. Probability of occurrence of a deviation greater than the band shown due to random fluctuations is 4.6% (for a 2σ band).

Table 10  
GROUND-HOLD CONTROL SAMPLE STANDARD ERROR BANDS  $(2\sigma)^1$

M A T E R I A L	P R O P E R T Y					
	GROUND-HOLD MATERIALS	$\Delta$ WEIGHT GRAMS	$\Delta$ DENSITY PCF (G/CC)	TENSILE ULTIMATE, LB/IN (N/M)	TENSILE SHEAR PSI (N/M <sup>2</sup> )	COMPRESSIVE STRENGTH PSI (N/M <sup>2</sup> )
$\beta$ (Beta Glass)		$-.0006 \pm .0010$	-	$114 \pm 13$ $(2.00 \times 10^4 \pm .23 \times 10^4)$	-	-
NA (Narmco Adhesive 7343/7139)		$-.0011 \pm .0019$	-	-	$530 \pm 83$ $(3.65 \times 10^6 \pm .57 \times 10^6)$	-
GY (Goodyear Adhesive 4001/4004)		$-.0009 \pm .0012$	-	-	See Fig. 20	-
PF (Polyurethane Foam)		$-.0010 \pm .0008$	$-.02 \pm .016$ $(0.3 \times 10^{-4} \pm 2.5 \times 10^{-4})$	-	-	$36.3 \pm .61$ $(2.50 \times 10^5 \pm .04 \times 10^5)$

1. Probability of occurrence of a deviation greater than the band shown due to random fluctuations is 4.6% (for a  $2\sigma$  band).

Table 11  
MISCELLANEOUS CONTROL SAMPLE STANDARD ERROR BANDS (2σ)<sup>1</sup>

M A T E R I A L	P R O P E R T Y						
	MISCELLANEOUS MATERIALS	Δ WEIGHT GRAMS	TENSILE ULTIMATE PSI (N/M <sup>2</sup> )	TENSILE SHEAR PSI (N/M <sup>2</sup> )	PEEL LB/IN (N/M)	Δ ABSORBANCE	Δ EMITTANCE
V (Velcro Fasteners)		+ .0016 ± .0008	9.6 ± 2.9 (6.62x10 <sup>4</sup> ± 2.0x10 <sup>4</sup> )	23.6 ± 6.2 (1.63x10 <sup>5</sup> ± 4.3x10 <sup>5</sup> )	15 ± 6 (2627 ± 1051)	-	-
TFE (Teflon Film)		- .018 ± .030	(LB/IN) 46.5 ± 3.2 (N/M) (3.21x10 <sup>5</sup> ± 2.2x10 <sup>5</sup> )	-	-	-	-
TH-A (Thermatrol on Aluminum Substrate)		- .005 ± .007	-	-	-	+ .030 ± .012	- .002 ± .004
TH-F (Thermatrol on Fiberglass Substrate)		+ .113 ± .142	-	-	-	+ .008 ± .020	+ .002 ± .008
BL-A (Series 400 Black Paint on Aluminum Substrate)		- .01 ± .01	-	-	-	- .003 ± .008	+ .003 ± .005
BL-F (Series 400 Black Paint on Fiberglass Substrate)		- .002 ± .008	-	-	-	- .006 ± .002	+ .002 ± .003

1. Probability of occurrence of a deviation greater than the band shown due to random fluctuations is 4.6% (for a 2σ band).

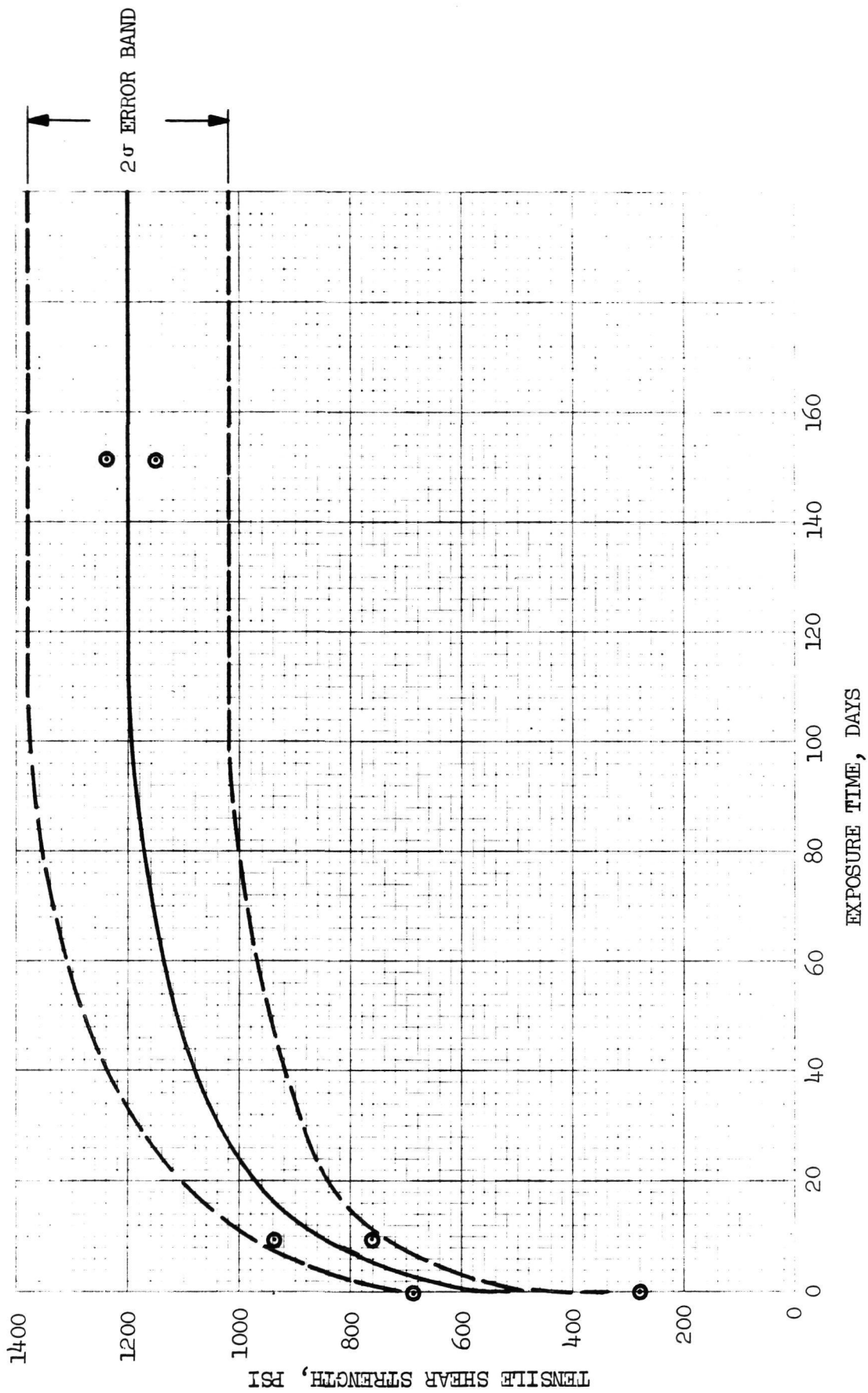


Fig. 20 Tensile Shear Strength for Goodyear Adhesive 4001/4004, Control Environment

5.2 SUMMARY OF POST EXPOSURE PROPERTY CHANGES (ENVIRONMENTS 2, 3, 5, 6, 7, 8)

Using the judgment criteria described in Section 5.1, percent changes were calculated for the post exposure test values that fall outside the error bands given previously in Tables 8 through 11 and Fig. 20. This percentage change was calculated as follows for the nondestructive tests <sup>1</sup> where both pre and post exposure measurements were made on the same specimen.

$$\% \text{ change} = \frac{(\Delta \bar{X} - \Delta \bar{X}_{\text{control}}) 100}{\bar{X}_{\text{pre}}} \quad (5-1)$$

For the destructive tests <sup>2</sup> the percentage change was calculated as follows.

$$\% \text{ change} = \frac{(\bar{X}_{\text{post}} - \bar{X}_{\text{control}}) 100}{\bar{X}_{\text{control}}}$$

(for samples where 0, 10 and 150 control data are combined)

or

$$\% \text{ change} = \frac{(\bar{X}_{\text{post}} - \bar{X}'_{\text{control}}) 100}{\bar{X}'_{\text{control}}} \quad (5-2)$$

(for Goodyear adhesive tensile shear samples where the control property values change with time)

where

- $\Delta \bar{X}$  = Post-exposure test value minus the pre-exposure value. (Mean  $\Delta$  value of the measurements taken.)
- $\Delta \bar{X}_{\text{control}}$  = Ten and 150 day control test values minus the zero day values. (Mean  $\Delta$  value of the measurements taken.)
- $\bar{X}_{\text{pre}}$  = Mean value of the pre-exposure test measurements taken.
- $\bar{X}_{\text{post}}$  = Mean value of the post-exposure test measurements taken.
- $\bar{X}_{\text{control}}$  = Mean value of the 0, 10 and 150 day control sample measurements.
- $\bar{X}'_{\text{control}}$  = Mean value of control sample measurement at time  $t$  corresponding to  $\bar{X}_{\text{post}}$ .

1. The nondestructive tests are weight, density, reflectance, absorbance, emittance.
2. The destructive tests are tensile ultimate, tensile shear, peel, compressive strength and layer density.

These percentage change results are presented in Tables 12 through 15 for the radiation shields, spacer materials, ground hold materials and miscellaneous materials. A discussion of these results is provided in Section 6.

### 5.3 OXYGEN AND FLUORINE IGNITION TEST RESULTS (ENVIRONMENTS 8a AND 8b)

The test materials were placed in stainless steel test chambers, the pressure was lowered to  $10^{-6}$  torr ( $1.33 \times 10^{-4}$  n/m<sup>2</sup>) and the chamber was back-filled to 760 torr ( $10^5$  n/m<sup>2</sup>) with GO<sub>2</sub> (Environment 8a) or GF<sub>2</sub> (Environment 8b) as described previously in Section 3.8. If ignition occurred, the ignition pressure was noted as well as the maximum pressure spike. For the oxygen tests, no materials ignited and a post test inspection revealed no visual changes in any of the materials. The results of the fluorine ignition tests are provided in Table 17. Tests were repeated on four materials where unexpected results occurred the first time. Identical results were obtained with single aluminized Kapton, single Goldized Kapton and Goodyear adhesive. The Beta glass cloth showed no reaction for the first test and a mild reaction for the repeat tests. A discussion of these results is provided in Section 6.

### 5.4 SUMMARY OF OUTGASSING TEST RESULTS (ENVIRONMENTS 2b, 2c, 2d and 4)

The outgassing test results for environments 2b, 2c, and 4 are provided in tabular and graphical form in Volume II.

For environment 2d, it was suspected in advance that no outgassing would be detected with the sample at 140°R (78°K). To verify this hypothesis, a test was conducted using a combined sample of ten materials with the highest outgassing rates as noted from prior test results. Even with such a large sample, the chamber pressure with the sample in place did not exceed the pressure for the empty chamber. In fact, the pressure with the sample was slightly lower, because of the substantial area for cryopumping. Consequently, the only information obtained for environment 2d is that the

# PERCENT CHANGE IN PROPERTIES FOR EXPOSED MULTILAYER INSULATION RADIATION SHIELD MATERIALS

(ALL MATERIALS 0.25 MIL ( $6.4 \times 10^{-3}$  MM))

W	=	WEIGHT
AD	=	COATING ADHESION (% REMOVED)
F <sub>b</sub>	=	FLEXIBILITY
F <sub>t</sub>	=	TENSILE ULTIMATE
ε <sub>t</sub>	=	NEAR NORMAL EMITTANCE
ρ <sub>s</sub>	=	SOLAR REFLECTANCE
1.		
		P. P. = PARTIAL PRESSURE
		F <sub>2</sub> P. = VARIES FROM 0 TO 760 TORR
		- = NO CHANGE WITHIN STANDARD
		ERROR BAND ( $\pm \sigma$ )
		2.
		D = SPECIMEN DETERIORATED IN ENVIRONMENT
		TO EXTENT POST-ENVIRONMENT TESTS COULD
		NOT BE PERFORMED
		L/O = EMITTANCE VALUES SEPARATED FOR
		EACH SIDE "L" SIDE AND "O" SIDE
		BLANK ENTRY INDICATES DATA TAKEN
		7. WAS CONSIDERED INVALID
		TO EXTENT POST-ENVIRONMENT TESTS COULD
		NOT BE PERFORMED
		3.
		4.
		5.
		6.
		7.



TABLE 13

## PERCENT CHANGE IN PROPERTIES FOR EXPOSED MULTILAYER INSULATION SPACER MATERIALS

PROPERTY ENVIRONMENT	EXPOSURE PARAMETERS		SILK NET (SN)			NYLON NET (NN)			DACRON NET (DN)			TISSUGLAS (TG)		
	O <sub>2</sub> /F <sub>2</sub> P, P <sub>2</sub> TORR	TEMP TIME R° K HR	W	LD	F <sub>t</sub>	W	LD	F <sub>t</sub>	W	LD	F <sub>t</sub>	W	LD	F <sub>t</sub>
SIMULATED ATMOSPHERIC ENVIRONMENTS (AT AMBIENT PRESSURE)														
200° F (93° C) 40% R. H.	3a	24	-1.5	-42	—	-1.5	—	—	-2	—	—	-2	+17	—
		72	-1.7	-42	—	-2.6	—	—	-7	—	—	-1	—	—
		240	-1.9	-40	-35	-3.9	—	+40	-5.8	—	—	—	+15	—
95% R. H. AT 95° F (35° C)	5a	12	—	-44	—	—	—	—	-9	—	—	—	—	—
		24	-5.4	-38	—	—	—	—	—	—	—	—	—	—
		72	+1.4	—	—	—	—	—	-6	—	—	+2	—	—
95% R. H. /SALT AIR AT 95° F (35° C)	6a	12	+9.0	—	—	+8	—	—	+2.5	—	—	+14	-17	—
		24	+5.6	—	-15	+13	—	—	+3.4	—	—	+19	-35	—
		72	+4.1	—	-13	+12	—	—	+4.3	—	—	+21	-51	-81
WATER IMMERSION AT 70° F (21° C)	7a	0.5	-17	—	—	—	—	—	-8	—	—	D	D	D
		2	-17	—	—	—	+16	—	-1.1	—	D	D	D	D
		24	-22	—	—	—	+16	—	—	—	D	D	D	D
95% R. H. /F <sub>2</sub> AT 95° F (35° C)	8g	10 <sup>-3</sup>	—	—	—	—	—	—	—	—	—	-2	—	—
		760 <sup>3</sup>	D <sup>5</sup>	D	D	D	D	D	D	D	D	D	D	D
		4	—	—	—	—	—	—	—	—	—	—	—	—
SIMULATED ORBIT ENVIRONMENTS														
GASEOUS FLUORINE AT 70° F (21° C)	8e	10 <sup>-3</sup>	—	-43	—	-2.4	—	D	+7	—	-26	-1.6	-22	—
		10 <sup>-3</sup>	—	-35	-13	—	—	—	+2.6	—	-20	-1.3	—	-70
		3600	—	—	—	—	—	—	—	—	—	—	—	—
GASEOUS OXYGEN AT 70° F (21° C)	8c	10 <sup>-3</sup>	—	-32	—	-5	—	—	—	—	—	—	—	—
		10 <sup>-3</sup>	-8	—	—	-7	—	—	—	—	—	+2	—	—
		3600	—	—	—	—	—	—	—	—	—	—	—	—
VACUUM, ≤ 10 <sup>-6</sup> TORR	2a	660 (366)	24	-35	—	-2.3	—	—	—	—	—	-2	—	—
		660 (366)	240	-31	—	-3.3	—	-24	-1.6	—	—	-1	—	—
		530 <sup>+</sup> (295)	240	—	-17	-1.6	+16	—	-5	—	—	—	-27	—
		140 (78)	240	—	—	—	+22	—	—	—	—	-1	—	—
		37 (21)	240	—	—	—	—	—	-1.5	—	+16	+2	+17	—
		240	—	—	—	—	—	—	—	—	—	—	—	—

1. W = WEIGHT

LD = FREE STANDING LAYER DENSITY AFTER  
COMPRESSION TO ONE-HALF THE INITIAL  
THICKNESS (AS COMPARED TO THE CON-  
TROL SAMPLE AFTER THE IDENTICAL  
COMPRESSION PROCEDURE)

F<sub>t</sub> = TENSILE ULTIMATE

2. P. P. = PARTIAL PRESSURE

3. F<sub>2</sub> = PARTIAL PRESSURE VARIES FROM 0 TO 760 TORR

4. — = NO CHANGE WITHIN STANDARD ERROR BAND

(±2 σ)

5. D = SPECIMEN DETERIORATED IN ENVIRONMENT TO  
EXTENT POST-ENVIRONMENT TESTS COULD NOT  
BE PERFORMED

6. BLANK ENTRY INDICATES DATA TAKEN  
WAS CONSIDERED INVALID



TABLE 14

## PERCENT CHANGE IN PROPERTIES FOR EXPOSED GROUND-HOLD INSULATION MATERIALS

PROPERTY <sup>1</sup> ENVIRONMENT	EXPOSURE PARAMETERS		BETA GLASS CLOTH (B)		NARMCO 7343/7139 ADHESIVE (NA)		GOODYEAR PLOBOND 4001/4004 ADHESIVE (GY)		POLYURETHANE FOAM, 2 PCF (PF)		
	O <sub>2</sub> OR F <sub>2</sub> P.P. 2 TORR	TEMP °R (°K)	TIME, HR	W	F <sub>t</sub>	W	F <sub>s</sub>	W	F <sub>s</sub>	W	F <sub>c</sub>
SIMULATED ATMOSPHERIC ENVIRONMENTS (AT AMBIENT PRESSURE)											
200 ° F (93° C) 40% R. H.	3a		24	—	+25	—	+34	—	-20	-2.0	-27
	3b		72	-5	+28	—	+52	—	—	-2.9	-31
	3c		240	+3	+45	—	+94	—	—	-4.7	-33
95% R. H. AT 95° F (35° C)	5a		12	-9	—	—	—	—	-16	—	-2
	5b		24	-1.9	—	—	—	—	-47	—	-4
	5c		72	—	—	—	—	—	-47	—	-3
95% R. H./SALT AIR AT 95° F (35° C)	6a		12	-2.0	—	+4	—	+2	-38	+4.4	-7.2
	6b		24	-8	+31	-3	—	+1	-38	+3.8	-8.0
	6c		72	+2.0	—	+1	-20	.1	-39	+7.4	+2
WATER IMMERSION AT 70° F (21° C)	7a		0.5	—	-22	—	—	—	-59	—	-2
	7b		2	—	—	—	—	—	-52	—	-3
	7c		24	—	—	—	—	—	-48	—	-1
95% R. H./F <sub>2</sub> AT 95° F (35° C)	8g	10 <sup>-3</sup>	4	—	+28	—	—	—	-25	-5	-2
	8h	760 <sup>3</sup>	4	+1.1	-59	D <sup>5</sup>	D	+0.04	-30	D	D
SIMULATED ORBIT ENVIRONMENTS											
GASEOUS FLUORINE 8e AT 70° F (21° C)	8e	10 <sup>-3</sup>	100	—	+30	—	—	—	+28	-5	-2
	8f	10 <sup>-3</sup>	3600	-2	+50	+0.005	+25	—	-26	-2.3	-3
	8c	10 <sup>-3</sup>	100	-3	—	—	+18	—	-30	-2.0	-4
GASEOUS OXYGEN AT 70° F (21° C)	8d	10 <sup>-3</sup>	3600	-6	—	—	+17	—	—	-2.6	-3
	2a		660 (366)	-4	—	—	+55	—	—	-5.5	-23
	2b		660 (366)	-8	—	—	+91	—	+20	-5.4	-25
VACUUM, ≤ 10 <sup>-6</sup> TORR	2c		530 (295)	-4	—	—	+28	—	-32	-2.2	-4
	2d		140 (78)	—	—	—	—	—	-39	—	-1
	2e		37 (21)	—	—	—	—	—	-42	—	-3

1. W = WEIGHT  
 F<sub>s</sub> = TENSILE SHEAR  
 F<sub>t</sub> = TENSILE ULTIMATE  
 ρ = DENSITY  
 F<sub>c</sub> = COMPRESSION ULTIMATE
2. P, P. = PARTIAL PRESSURE  
 3. F<sub>2</sub> = PARTIAL PRESSURE VARIES FROM 0 TO 760 TORR  
 4. — = NO CHANGE WITHIN STANDARD ERROR BAND (±2σ)
5. D = SPECIMEN DETERIORATED IN ENVIRONMENT TO EXTENT POST-ENVIRONMENT TESTS COULD NOT BE PERFORMED.

TABLE 15

## PERCENT CHANGE IN PROPERTIES FOR EXPOSED MISCELLANEOUS INSULATION MATERIALS

PROPERTY <sup>1</sup> ENVIRONMENT	EXPOSURE PARAMETERS		POLYESTER VELCRO FASTENERS(V)			TFE- TEFLON FILM (TFE)		THERMATROL PAINT ON ALUMINUM (TH-A)			THERMATROL PAINT ON FIBERGLASS (TH-F)			SERIES 400 BLACK PAINT ON ALUMINUM (BL-A)			SERIES 400 BLACK PAINT ON FIBERGLASS (BL-F)					
	O <sub>2</sub> OR F <sub>2</sub> P.P. 2 TORR	TEMP °R(K) HR	W	F <sub>t</sub>	F <sub>s</sub>	F <sub>p</sub>	W	F <sub>t</sub>	F <sub>b</sub>	W	AD	ε	α	W	AD	ε	α	W	AD	ε	α	
SIMULATED ATMOSPHERIC ENVIRONMENTS (AT AMBIENT PRESSURE)																						
200° F (93° C) 40% R. H.		24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		72	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
		240	-	+58	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
95% R. H. AT 95° F (35° C)		12	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
		72	+2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
95% R. H./SALT AIR AT 95° F (35° C)		12	+1.3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		24	+2.1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		72	+11.2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
WATER IMMERSION AT 70° F (21° C)		0.5	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
95% R. H./F <sub>2</sub> AT 95° F (35° C)		4	+1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		4	D <sup>5</sup>	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
SIMULATED ORBIT ENVIRONMENTS																						
GASEOUS FLUORINE AT 70° F (21° C)		100	+3	+51	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		3600	+6	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
GASEOUS OXYGEN AT 70° F (21° C)		100	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		3600	+2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
2a		660 (366)	24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		660	240	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		530 (295)	240	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		140 (78)	240	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
2b		37 (21)	240	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
2c			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
2d			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
2e			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

1. W = WEIGHT  
F<sub>b</sub> = FLEXIBILITY  
α = SOLAR ABSORBANCE  
F<sub>t</sub> = TENSILE ULTIMATE  
AD = PAINT ADHESION  
(% REMOVED)
2. P.P. = PARTIAL PRESSURE  
3. F<sub>2</sub>P.P. = VARIES FROM 0 TO 760 TORR  
4. - = NO CHANGE WITHIN STANDARD ERROR  
BAND (±2σ)  
5. D = SPECIMEN DETERIORATED IN ENVIRONMENT  
TO EXTENT POST-ENVIRONMENT TESTS COULD  
NOT BE PERFORMED
- F<sub>s</sub> = TENSILE SHEAR  
ε<sub>s</sub> = NEAR NORMAL EMISSANCE  
F<sub>p</sub> = T-PEEL STRENGTH

Table 16  
FLUORINE IGNITION TEST RESULTS (ENVIRONMENT 8B)

SPECIMEN (2 EA)	POST TEST INSPECTION	IGNITION PRESS PSIA (N/M <sup>2</sup> )	PRESS SPIKE PSIA (N/M <sup>2</sup> )
Single Alumin- ized Mylar	No sample solid material left. A little black soot and aluminum de- posit left in can.	14.1 (9.7 x 10 <sup>4</sup> )	44.5 (3.07 x 10 <sup>5</sup> )
Double Alumin- ized Mylar	No damage or discoloration observed.	No ignition	-
Single Alumin- ized Kapton*	No damage or discoloration observed.	No ignition	-
Single Goldized Mylar	No sample solid material left. A little gold dust deposited on can surfaces.	13.9 (9.6 x 10 <sup>4</sup> )	32.6 (2.25 x 10 <sup>5</sup> )
Double Goldized Mylar	No damage or discoloration observed.	No ignition	-
Single Goldized Kapton*	No sample solid material left. Gold dust deposited on can surfaces.	7.3 (5.0 x 10 <sup>4</sup> )	33.2 (2.29 x 10 <sup>5</sup> )
Silk Net (with sizing)	No damage or discoloration observed.	No ignition	-
Silk Net (with sizing removed)	Black residue found in the bottom of the can.	Pressure data inadvertently not recorded but ignition pressure was < 14.7 psi (10 <sup>5</sup> n/m <sup>2</sup> )	
Nylon Net	No sample solid material left. Some black deposits observed on can surfaces.	3.7 (2.6 x 10 <sup>4</sup> )	25.0 (1.72 x 10 <sup>5</sup> )
Tissuglas	No sample solid material left. Some black deposits found in can.	6.0 (4.1 x 10 <sup>4</sup> )	16.2 (1.12 x 10 <sup>5</sup> )
Dacron Net	No sample solid material left. Some black deposits found in can.	15.5 (1.07 x 10 <sup>5</sup> )	31.6 (2.18 x 10 <sup>5</sup> )
Beta Glass Cloth	No damage or discoloration observed.	No ignition	-
Beta Glass Cloth (Repeat Test)	Slight brownish discoloration.	Pressure data inadvertently not recorded but ignition pressure was < 14.7 psi (10 <sup>5</sup> n/m <sup>2</sup> )	
Narmco Adhesive	Material partially burned. Extreme discoloration (black) where fluorine flowed onto sample.	1.1 (7.6 x 10 <sup>3</sup> )	18 (1.24 x 10 <sup>5</sup> )
Goodyear Adhesive*	No damage or discoloration observed.	No ignition	-
Polyurethane Foam	No sample solid material left.	2.95 (2.03 x 10 <sup>4</sup> )	23.5 (1.62 x 10 <sup>5</sup> )
Velcro Fasteners	Black solid material fragments remained.	4.6 (3.17 x 10 <sup>4</sup> )	36 (2.48 x 10 <sup>5</sup> )
Teflon	No damage or discoloration	No ignition	-
Thermatrol Paint on Aluminum	White particles found in can. Sample sheets have burned spots.	9.2 (6.3 x 10 <sup>4</sup> )	47 (3.24 x 10 <sup>5</sup> )
Thermatrol Paint on Fiberglass	Fiberglass partly burned and charred.	6.0 (4.1 x 10 <sup>4</sup> )	33.2 (2.29 x 10 <sup>5</sup> )
Black Paint on Aluminum	Black soot found on sample. One sample shows indication of heat.	1.39 (9.6 x 10 <sup>3</sup> )	20.5 (1.41 x 10 <sup>5</sup> )
Black Paint on Fiberglass	Black soot in can. Fiberglass partly burned and charred.	0.5 (3.4 x 10 <sup>3</sup> )	37.4 (2.58 x 10 <sup>5</sup> )

\* Test was repeated with same results

outgassing rate for all samples at 140°R (78°K) is effectively zero.

The tabular listing for the remaining environments 2b, 2c and 4 (shown in Volume II) includes the pre-exposure sample weight, the total nominal exposed surface area and the outgassing rate with the fraction of each gas species present at discrete points in time. On the opposite page in Volume II, the outgassing data are presented in graphical form as a function of time. The same ordinate and abscissa scales are used for all area-based data and for all weight-based data to facilitate visual comparison.

A discussion of these results is provided in Section 6.

## Section 6

### DISCUSSION OF RESULTS

For all the test data taken (except for the in-situ propellant ignition, 8a and 8b, and outgassing measurements), the pre and post-exposure tests were performed in a 70°F (21°C) ambient laboratory environment. The post-exposure tests were performed as soon as possible after the specimens were removed from the specified environment, usually within one day. Nevertheless, there is a time period between the exposure and the test in which property changes produced by the environment could "anneal" and not be detected. Performing the tests within the environment would eliminate this potential problem but would have increased the scope of the program enormously and was not considered justified. Property values where this "annealing" effect could be real includes weight, density (of foam in vacuum), emittance, reflectance and absorbance.

Also, in interpreting the test results shown in the previous section, it should be borne in mind each percentage change value shown in Tables 12 through 15 represents the mean value of a maximum of two data points. Due to this limited amount of statistical data, if a small property change was noted for one of the shorter exposure times but the trend was not verified by consistent changes at longer exposure times, the change was not considered significant. If the property change was (1) large at the short exposure time or (2) was at the longest exposure time or (3) if consistent changes were noted with increasing exposure times, then the property change was attributed to the effects of the environmental exposure and considered significant.

Using these general guidelines for interpreting the data, significant property changes were analyzed and are summarized within the four major material groupings that follow in Sections 6.1 through 6.4. (Materials which exhibit no significant property changes are summarized in Section 6.6.) Outgassing test results are discussed separately in Section 6.5. Recommendations on material usage in different environments conclude this section in 6.6.

## 6.1 RADIATION SHIELD TEST TRENDS

The significant test trends for radiation shields are discussed as a function of different measured material property values for those materials that survived the environmental exposures. The materials that deteriorated in the post-exposure environments to the extent certain measurements could not be made include all the aluminized specimens in the 72-hour salt air exposure, 6c,<sup>1</sup> (the aluminum coating was removed) and S-A-M, S-G-M and S-G-K specimens in the moist air/fluorine environment, 8h (the specimens were destroyed). Significant property changes noted in the remaining applicable exposure conditions are discussed below for single aluminized Mylar (S-A-M), double aluminized Mylar (D-A-M), single aluminized Kapton (S-A-K), single goldized Mylar (S-G-M), double goldized Mylar (D-G-M) and single goldized Kapton (S-G-K).

### 6.1.1 Weight

Weight changes for all the radiation shields following all the environments were less than 3% with the following exceptions:

- ° A 7 to 17% weight increase following the salt air environment (6) due to salt deposition.
- ° A 5% increase for D-A-M and a 6% increase for S-A-K following the moist air/fluorine environment (8h) apparently due to a chemical reaction.

### 6.1.2 Adhesion

Before the adhesion test was performed, changes in the visual appearance of the samples after exposure was noted as described in the following paragraph. Following the 72-hour, 95% R.H. exposure (5c), a faint pattern developed in

---

1 Refers to the exposure environment shown in Section 3

the aluminum coatings as shown in Fig. 21. This pattern plus the appearance of pinholes in the aluminum coating became more pronounced in the 95% R.H./salt air environment going from the 12-hour exposure to the 24-hour exposure; the aluminum coating disappeared following the 72-hour exposure as shown in Fig. 22. Following the same 72-hour exposure (6c), some minute pinholes were observed on the gold coatings as well. No coating damage was noted in any of the other environments (where the materials survived).

Results of the coating adhesion test follows:

- ° Aluminum adhesion to both Mylar and Kapton is degraded by the salt air environment after 24 hours (6c); the coating is completely removed after 72 hours.
- ° Gold adhesion to Mylar is degraded by high temperature (3), high humidity (5), and salt air (6) environments. Gold adhesion to Kapton is degraded by high humidity (5) and to a lesser extent by gaseous fluorine/moist air (8g), low pressure gaseous fluorine (8f) and low pressure gaseous oxygen (8d) environments.

#### 6.1.3 Flexibility

No changes occurred in coating or substrate flexibility for any of the surviving materials following any of the environmental exposures.

#### 6.1.4 Ultimate Tensile Strength

S-A-M showed no significant changes in any of the environments while the tensile strength of D-A-M and S-A-K both decreased in the moist air/fluorine environment, -19 to -34% (8h). S-A-K also showed a decrease of -20% in the low pressure fluorine environment (8f).

#### 6.1.5 Near Normal Emittance

All the aluminized materials showed degraded (increased) emittance values of





S-A-K



S-A-M

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D-A-M

Fig. 21 Aluminized Films After a 72-Hour High Humidity Exposure (5c)

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S-A-M



S-A-K



D-A-M

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Fig. 22 Aluminized Films After a 72-Hour Salt Air Exposure (6c)

from 10 to 52% following the 24-hour salt air exposure (6b); S-A-M's emittance also degraded +58% following the 72-hour high humidity exposure (5c) while the moist air/fluorine environment (8h) significantly degraded the emittance value of D-A-M 65%.

All the gold-coated materials showed a significant improvement (decrease) in emittance of from -29 to -52% following the 240-hour high temperature vacuum exposure (2b); evidently, this environment "cleaned" the gold surfaces.

#### 6.1.6 Solar Reflectance

The aluminized materials S-A-M and S-A-K showed contradictory trends for solar reflectance following exposure to several different environments, with S-A-M degrading (decreasing) and S-A-K improving (increasing). The affected environments with the maximum percent changes are shown below.

	<u>S-A-M</u>	<u>S-A-K</u>
Salt Air (6b)	- 9	+ 7
Water Immersion (7)	- 3	+15
Moist Air/Fluorine (8g)	- 3	+17
Low Pressure Fluorine (8e)		+ 7
Low Pressure Oxygen (8f)		+12
High Temperature/Vacuum (2a, b)	- 6	+ 8
Low Temperature/Vacuum (2d, 2e)		+12

Since the solar reflectance measurement is made on the non-aluminized side only, (1) the exposed Mylar and Kapton may be affected differently by the respective environments, or (2) the control sample values may be off slightly and are introducing a systematic error into the calculations.

None of the surviving gold coated samples showed significant changes in solar reflectance following any of the environmental exposures.

#### 6.1.7 $\text{GF}_2$ Ignition (8b)

The double coated Mylar substrates (aluminum and gold) did not ignite in a

760 torr ( $10^5$  n/m<sup>2</sup>) gaseous fluorine environment. Apparently, the thin metal coating provides reasonable protection for short periods of exposure (hours).

For the single coated aluminized films, S-A-K did not ignite while S-G-K did ignite. The tests were repeated with the same results; in addition, S-A-K survived a one atmosphere moist air/fluorine environment (8h). The reason for this anomaly between the behavior of S-A-K and S-G-K is not understood since each specimen has one unprotected Kapton side.

#### 6.1.8 GO<sub>2</sub> Ignition (8a)

None of the shields ignited in a 760 torr ( $10^5$  n/m<sup>2</sup>) gaseous oxygen environment. (The test setup did not include an ignition source.)

#### 6.1.9 Outgassing

Outgassing rates for the radiation shields were normalized at 1, 10 and 100 hours in Table 17 with the lowest outgassing rate shown first.

At 200°F (93°C), the Kapton materials outgas less than Mylar, the double coated materials outgas less than single coated materials and the gold coatings outgas less than the aluminum coatings.

The rankings of the shields from one environment to another show no consistently best or worst material from the point of outgassing, but certain trends are evident. Single aluminized Mylar appears to have the highest rate. The single goldized Mylar and single goldized Kapton hold eleven out of fourteen top rankings. Also, single aluminized Kapton and single goldized Kapton appear in the upper third of the rankings in fifteen out of a possible twenty-four instances even though there are twice as many Mylar shields as Kapton shields listed. Hence, although there are specific exceptions, it is concluded overall that Kapton outgasses less than Mylar, and as would be anticipated, the gold film outgasses less than the aluminum film.

Table 17

## NORMALIZED OUTGASSING RATES FOR RADIATION SHIELDS IN VACUUM

	<u>1 Hour</u>		<u>10 Hours</u>		<u>100 Hours</u>	
<u>Environment 2b, 200°F (93°C)</u>	SGK	1.0 —	SGK	1.0 —	SGK	1.0
	SAK }	1.4 —	SAK }	—	SAK }	5.0
	DGM }	—	DGM }	4.6 —	DGM }	—
	DAM	3.3 —	DAM	—	DAM	—
	SGM	4.0 —	SAM	8.5 —	SAM	10.0
	SAM	4.2 —	SGM	11.5 —	SGM	11.4
<u>Environment 2c, 70°F (21°C)</u>	SGM	1.0 —	SGM	1.0 —	SGM	1.0
	SAK	3.3 —	SGK	3.6 —	SGK	—
	DAM	20 —	DAM	7.3 —	DAM	—
	DGM	40 —	SAK	13 —	SAK	12
	SGK	50 —	DGM	58 —	DGM	16
	SAM	100 —	SAM	326 —	SAM	104
<u>Environment 4a, 6 hours at 200°F (93°C) followed by a 4-hour nitrogen purge*</u>	SGK	1.0 —	DAM	1.0 —	DAM	1.0
	SAK	1.6 —	SAK	2.1 —	SAM	2.4
	DAM	1.7 —	SAM	2.4 —	SAK	4.5
	DGM	3.3 —	SGK	3.5 —	DGM	7.5
	SAM	8.3 —	DGM	4.7 —	SGK	8.7
	SGM	23 —	SGM	47 —	SGM	150
<u>Environment 4b, 6 hours at 200°F (93°C) followed by a 4-hour helium purge*</u>	SGK	1.0 —	SGM	1.0 —	SGM	1.0
	SGM	1.9 —	SGK	1.1 —	SGK	1.2
	DGM	3.0 —	DGM	2.5 —	DAM	1.6
	SAK	48 —	DAM	4.9 —	DGM	2.2
	SAM	100 —	SAM	29 —	SAM	26
	DAM	200 —	SAK	55 —	SAK	122

\* See Section 6.5 for a discussion of the validity of these test data on an absolute scale.

## 6.2 SPACER MATERIAL TEST TRENDS

The significant test trends for spacer materials are discussed in this section as a function of different measured material property values for those materials that survived their respective environmental exposures. (All four spacer materials were destroyed in the moist air/fluorine environment (8h)<sup>1</sup> while Tissuglas disintegrated in the water immersion environment (7).) Significant property changes noted in the remaining applicable exposure conditions are discussed below for silk net with sizing (SN), nylon net (NN), dacron net (DN) and Tissuglas (TG).

### 6.2.1 Weight

All the spacers showed weight increases following the salt air environment (6) due to salt deposition with DN showing the smallest increase and TG the largest. SN, NN and DN showed increasing weight losses with time following the high temperature exposure at 200°F (93°C) for both the ambient pressure (3) and the high vacuum (2a, 2b) exposures. Water immersion (7) of SN dissolved most of the sizing, leaving the SN in a limp condition with up to a 22% weight reduction. In the low pressure gaseous fluorine environment (8e, 8f) DN gained weight (+2.6%) while TG lost weight (-1.3%). The TG weight losses are approximately an order of magnitude higher than those in vacuum (2) alone; therefore, the weight loss cannot be attributed to outgassing only and may be due to chemical formation and loss of  $\text{SiF}_4$  gas. The DN weight change is consistent with weight gains noted later for Velcro fasteners (both materials are polyesters).

### 6.2.2 Free Standing Layer Density After Compression to One-Half the Initial Thickness

DN showed no layer density changes (as compared to a control sample under-

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1. Refers to the environmental exposure shown in Section 3.

going the same compression cycle) after any of the environmental exposures. NN was nearly as good as DN showing only an increase of +16% (more layers/unit thickness) after the water immersion tests (7).

TG showed an increase of +15% after the 200°F (93°C) exposure (3) and a significant decrease of -51% (less layers/unit thickness) after the salt air exposure (6). Noting the large weight increases measured for Tissuglas following environment 6 (due to salt deposition), it appears this quantity of salt coating the Tissuglas fibers made the layers more "rigid". This coating tentatively explains why the layer density decreased.

SN (with sizing) showed significant layer density decreases with high temperature, -40%, (3), a combination of high temperature and vacuum, -31%, (2a, 2b), high humidity, -38%, (5) and low pressure fluorine, -35%, (8e, 8f) and oxygen, -32%, (8c, 8d) exposures. Evidently, high temperature, moisture and vacuum all tend to rigidize the water soluble sizing (similar to the action of starch in an ironed shirt) making the layers less pliable; consequently, less layers per inch are measured than with a limp fabric. This hypothesis is borne out to some extent by examining the SN water immersion (7) results. No change in layer density was noted following the immersion plus air drying yet significant decreases in weight were measured, up to -22%. With the sizing nearly removed by the water, the SN remained limp and the layer density properties did not change much from the SN with sizing.

### 6.2.3 Ultimate Tensile Strength

NN showed a large strength increase of 40% after exposure to high temperature (3) while DN's strength decreased around -23% following a low pressure fluorine exposure (8e, 8f). Otherwise, these two spacers showed no strength changes following exposure in the other environments.

TG's strength decreased markedly following both the salt air, -81%, (6) and

low pressure gaseous fluorine exposures, -70%, (8e, 8f). The low absolute strength of TG was found to vary greatly from sample to sample; this large scatter was reflected in the wide error band calculated for the control samples. This wide error band in turn may have obscured strength test trends in some of the other environments.

SN showed strength decreases following exposure to high temperature, -35%, (3), salt air, -13%, (6), and low pressure gaseous fluorine, -13%, (8e, 8f) environments. This decrease cannot be attributed to effects on the sizing material since no strength changes were noted following water immersion (7) when nearly all the sizing was removed.

#### 6.2.4 $\text{GF}_2$ Ignition (8b)

Both NN and TG ignited in a 760 torr ( $10^5 \text{ n/m}^2$ ) gaseous fluorine environment. DN ignited at a slightly higher pressure of 800 torr ( $1.07 \times 10^5 \text{ n/m}^2$ ). DN also ignited in a moist air/fluorine environment (8h) confirming its susceptibility to fluorine or hydrofluoric acid attack. SN (with the fire retardent sizing) was not attacked by fluorine at 760 torr ( $10^5 \text{ n/m}^2$ ). However, when the sizing was removed, the SN was destroyed by fluorine exposure. The role of the sizing in protecting the SN from fluorine attack was further confirmed by the moist air/fluorine exposure (8h). The moist air apparently removed enough sizing to where the fluorine or hydrofluoric acid could attack and destroy the SN.

#### 6.2.5 $\text{GO}_2$ Ignition (8a)

None of the spacers ignited in a 760 torr ( $10^5 \text{ n/m}^2$ ) gaseous oxygen environment. (The test setup did not include an ignition source.)

#### 6.2.6 Outgassing

Outgassing rates for the spacers were normalized at 1, 10 and 100 hours in



Table 18 with the lowest outgassing rate shown first.

Over the temperature range 70°F (21°C) to 200°F (93°C), DN has the lowest overall outgassing rates. On the other hand silk net has the lowest overall outgassing rates for the preconditioning environments (4a and 4b) although DN was not too much higher than SN.

Also, Tissuglas has a surprisingly low outgassing rate when pumped at ambient temperature. However, when heated, a very large quantity of gas is evolved. Apparently there is a considerable amount of gas absorbed in the as-received Tissuglas, but it is tightly bonded and does not desorb easily without the application of heat.

### 6.3 GROUND-HOLD MATERIAL TEST TRENDS

The significant test trends for ground-hold materials are discussed in this section as a function of different measured material property values for those materials that survived their respective environmental exposures. (Two materials which did not survive the moist air/fluorine environment (8h)<sup>1</sup> are Narmco adhesive and polyurethane foam.) Significant property changes noted in the remaining applicable environments are described below for

- ° Beta glass cloth (B),
- ° Narmco 7343/7139 adhesive (NA),
- ° Goodyear 4001/4004 adhesive (GY), and
- ° polyurethane foam (PF).

#### 6.3.1 Weight

Only polyurethane foam exhibited significant weight changes of the four ground-hold materials. Weight loss following a 200°F (93°C) environment was slightly higher (-5.5%) when combined with a vacuum environment (2a, 2b)

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1 Refers to the environmental exposure shown in Section 3

Table 18

## NORMALIZED OUTGASSING RATES FOR SPACERS IN VACUUM

	<u>1 Hour</u>		<u>10 Hours</u>		<u>100 Hours</u>	
<u>Environment 2b, 200°F (93°C)</u>	DN	1.0	SN	1.0	SN	1.0
	NN	12.0	DN	1.2	DN	1.5
	SN	20.	NN	3.3	NN	2.5
	TG	44.	TG	58.	TG	55
<u>Environment 2c, 70°F (21°C)</u>	TG	1.0	TG	1.0	TG	1.0
	DN	1.3	DN	4.2	DN	10.
	SN	133.	SN	27.	SN	20.
	NN	266.	NN	150.	NN	60.
<u>Environment 4a, 6 hours at 200°F (93°C) followed by a 4-hour nitrogen purge*</u>	DN	1.0	SN	1.0	SN	1.0
	SN	1.3	DN	1.5	DN	1.5
	TG	2.9	TG	3.3	NN	23.
	NN	9.4	NN	16.	TG	57.
<u>Environment 4b, 6 hours at 200°F (93°C) followed by a 4-hour helium purge*</u>	SN	**	SN	**	SN	**
	NN	1.0	NN	1.0	NN	1.0
	TG	1.0	TG	1.0	TG	1.2
	DN	2.1	DN	1.4	DN	1.5

\* See Section 6.5 for a discussion of the validity of these test data on an absolute scale.

\*\* Measured outgassing rate is too small to quantify meaningfully.

than at ambient pressure, -4.7% (3). Weight losses following a low pressure gaseous fluorine or oxygen environment, -2.3 to -2.6%, (8c through 8f) appear to be comparable with weight losses in a vacuum environment (-2.2%) at the same ambient temperature (2c). The usual weight increase following the salt air exposure (6) is noted due to salt deposition.

#### 6.3.2 Density (PF)

Polyurethane foam density showed consistent decreases with increasing time following exposure at 200°F (93°C) for both ambient pressure (3) and vacuum (2a, b) exposures. The large density decreases noted (-25 to -33%) were due more to volume increases than due to weight losses. Minor density decreases (up to 4%) were also noted following exposure to high humidity (5) water immersion (7), moist air/fluorine (8g), low pressure oxygen/fluorine (8c through 8f) and vacuum (ambient to LH<sub>2</sub> temperature, 2c, 2d, 2e) environments.

#### 6.3.3 Ultimate Tensile Strength (B)

B showed large, unexplained, tensile strength increases following a 200°F (93°C) exposure, +45%, (3) and a low pressure gaseous fluorine exposure, +50%, (8e, f). A large strength decrease (-59%) was measured following the moist air/fluorine exposure (8h) probably due to hydrofluoric acid attack.

#### 6.3.4 Ultimate Shear Strength (NA, GY)

The room temperature-cured NA adhesive increased significantly in strength (91-94%) following a 200°F (93°C) exposure as expected both in vacuum (2a, b) and at ambient pressure (3). In addition, NA strength increases of approximately equal magnitude (17 to 28%) were noted at ambient temperature following exposure to vacuum (2c) low pressure gaseous fluorine (8e, f) and low pressure gaseous oxygen (8c, d).

GY adhesive showed significant strength decreases following exposure to high humidity, -47%, (5), salt air, -39%, (6), water immersion, -59%, (7), moist air/fluorine, -30%, (8g, h), low pressure gaseous fluorine, -26%.(8f) and vacuum at ambient to  $\text{LH}_2$  temperature, -32 to -42%, (2c, d, e). The sizable change in GY shear strength control values with time (shown previously in Fig. 20) casts some doubt on whether the above listed decreases are real or are due to a systematic error in the control measurements.

#### 6.3.5 Compression Strength (PF)

The decrease in compression strength for foam was greatest (-60%) following vacuum exposure (2a, b) at 200°F (93°C); the next greatest decrease (-46%) occurred following the 200°F (93°C) ambient pressure exposure (3). All the foam compression measurements were very consistent and reproducible. The remaining environments produced compression strength decreases in the following order:

° Vacuum, ambient temp (2c) and low pressure gaseous fluorine and oxygen (8c through 8f)	-14 to -17.0%
° High humidity (5)	-10.0%
° Salt air (6)	- 8.5%
° Water immersion (7)	- 5.8%
° Vacuum at $\text{LN}_2$ to $\text{LH}_2$ temp (2d, e)	- 4.0%

#### 6.3.6 $\text{GF}_2$ Ignition (8b)

Only GY and B did not ignite in a 760 torr ( $10^5 \text{ n/m}^2$ ) gaseous fluorine environment although a slight brownish discoloration was noted on one of the B samples.

#### 6.3.7 GO<sub>2</sub> Ignition (8a)

None of the ground-hold materials ignited in a 760 torr ( $10^5$  n/m<sup>2</sup>) gaseous oxygen environment. (The test setup did not include an ignition source.)

#### 6.3.8 Outgassing

Direct comparison of the outgassing rates for all the ground-hold materials is not useful (except for the two adhesives) since the materials are not candidates for the same generic function. The NA and GY adhesives have a roughly comparable outgassing rate at 660°R (366°K); at 530°R (294°K), the outgassing rate of NA continues to drop with time while GY plateaus after eight hours.

### 6.4 MISCELLANEOUS MATERIAL TEST TRENDS

The significant test trends for the miscellaneous materials are discussed in this section as a function of different measured material property values for those materials that survived their respective environmental exposures. (None of the materials except Teflon film survived the moist air/fluorine environment, 8h<sup>1</sup>). Significant property changes noted in the remaining applicable environments are described below for Velcro fasteners (V), Teflon film (TFE), Thermatrol paint on aluminum (TH-A), Thermatrol paint on fiberglass (TH-F), Series 400 black paint on aluminum (BL-A) and Series 400 black paint on fiberglass (BL-F).

#### 6.4.1 Weight

Weight changes for V are less than .3% following the prescribed environmental exposures except for low pressure gaseous fluorine, +.6%, (8e, f) and salt

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1. Refers to the environmental exposure shown in Section 3.

air, +11.2%, (6). The weight increase for the polyester V following the fluorine environment is consistent with the weight increase noted previously for the polyester DN. The salt air environment (6) weight increase is due to the salt deposition on the V.

TFE gained .2% in weight following the salt air environment and lost less than -.3% following any of the other environmental exposures.

TH-A showed no weight change following the 200°F (93°C) environment (3) yet TH-F showed an unexplained increasing weight with time for the same exposure, from .3% at 24 hours to +1.2% at 72 hours. The weight increase due to the salt air exposure is less than +.1% for either TH-A or TH-F. Weight changes for TH-A and TH-F are less than .3% following exposure to the other prescribed environments.

Weight losses for BL-A and BL-F were generally higher than those for TH-A and TH-F following exposure to the same environments; none of the BL-A or BL-F weight changes exceeded .9%. Following vacuum exposure at temperatures ranging from 660°R (365°K) to 37°R (21°K), BL-F always lost more weight than BL-A indicating the epoxy fiberglass substrate is losing weight as well as the black paint.

#### 6.4.2 Ultimate Tensile Strength (V, TFE)

V showed an increase in tensile strength following the high temperature environment (3) of +58%. However, this increase was not corroborated by the shear or peel test results following the same exposure where no changes took place. TFE showed tensile strength decreases of up to -20% in five different environments and tensile strength increases of +12% in the moist air/fluorine environment (8g, h). To verify if these changes are real, seven additional control sample specimens were run bringing the control sample total to 13 specimens. The new control sample results shifted the control sample mean less than .5% so apparently the tensile strength changes noted above are valid.

#### 6.4.3 Ultimate Shear and Peel Strength (V)

No changes were noted in shear or peel strength for V following exposure to the prescribed environments.

#### 6.4.4 Flexibility (TFE)

No changes in film flexibility for TFE were noted following exposure to the prescribed environments.

#### 6.4.5 Coating Adhesion (TH-A, TH-F, BL-A, BL-F)

The low pressure gaseous fluorine environment after 3600 hours of exposure degraded coating adhesion -6% for TH-A, -14% for TH-F, <.1% for BL-A and <.1% for BL-F. The vacuum exposure at  $LH_2$  temperature also degraded the coating adhesion of BL-A -.2%.

#### 6.4.6 Near Normal Emittance (TH-A, TH-F, BL-A, BL-F)

TH-A showed less than a 1.3% change in emittance following exposure to any of the prescribed environments and TH-F, BL-A and BL-F showed no significant changes.

#### 6.4.7 Solar Absorbance (TH-A, TH-F, BL-A, BL-F)

BL-A and BL-F showed no significant changes in solar absorbance following exposure to the prescribed environments. TH-A and TH-F showed contradictory trends on the solar absorbance results following the salt air exposure (6). TH-A absorbance values increased while TH-F values decreased. A possible explanation for this anomaly is the amount of salt deposited on the surface. TH-A showed up to a +.1% weight increase while TH-F showed no significant weight changes indicating the salt deposition layer is less for TH-F than TH-A. None of the other absorbance changes for TH-A and TH-F for other environments appeared to exhibit any significant trends.



#### 6.4.8 $\text{GF}_2$ Ignition (8b)

Only TFE did not ignite in a 760 torr ( $10^5 \text{ n/m}^2$ ) gaseous fluorine environment. These results were also verified in the moist air/fluorine environment (8h).

#### 6.4.9 $\text{GO}_2$ Ignition (8a)

None of the miscellaneous materials ignited in a 760 torr ( $10^5 \text{ n/m}^2$ ) gaseous oxygen environment. (The test setup did not include an ignition source.)

#### 6.4.10 Outgassing

Direct comparison of the outgassing rates for all the miscellaneous materials is not useful since the materials are not candidates for the same generic function. It is of interest to note, however, the outgassing rates of TH-A and BL-A are roughly comparable at  $660^\circ\text{R}$  ( $366^\circ\text{K}$ ) while TH-A has substantially lower outgassing rates than BL-A at  $530^\circ\text{R}$  ( $294^\circ\text{K}$ ) for up to approximately 130 hours.

### 6.5 QUALIFICATION OF OUTGASSING TEST TRENDS

By using previously published outgassing data plus common logic, the relative rankings between the outgassing rates in the different environments for each material could have been forecast prior to testing with a high degree of certainty as follows:

1. The rate in environment 2b at  $200^\circ\text{F}$  ( $93^\circ\text{C}$ ) should be higher and more persistent than in environment 2c (ambient temperature) because the initial quantity of sorbed gas should be the same and the addition of heat will increase the escape probability.
2. The rate in environment 2d at  $140^\circ\text{R}$  ( $78^\circ\text{K}$ ) should be essentially zero. In fact, the cold insulation should gain rather than lose sorbed gas due to condensation.

3. The rate in environments 4a and 4b (6 hours at 200°F (93°C) followed by a 4 hour ambient N<sub>2</sub> or He purge) should be essentially identical since
  - ° neither nitrogen nor helium will form chemical bonds with the samples,
  - ° neither gas forms strong physical bonds, and
  - ° the sample size is such that variation in Knudsen flow velocity through the pores will be negligible.
4. The rate in environments 4a and 4b should be substantially less than for 2c, if only because the samples will have been pre-pumped 6 hours, effectively initiating the 4a and 4b tests at the 6 hour point for 2c. The fact that the sample was heated during the first 6 hours should magnify the effect of the pre-pumping.

The evidence already available which would lead to the above listed trends is considerable so that if these effects are not observed in the present data, the experiment's accuracy must be questioned. It is important to emphasize this point because in the present experiments the expected quantitative relationships between 2b, 2c and 2d data were observed, whereas those between 4a, 4b and 2c data were not. Discussion of 4a and 4b should thus be centered upon why the expected behavior was not observed.

The environment 2c outgassing data plotted versus time show slopes on a log-log plot of between about -0.5 and -1.0, which is characteristic for ambient temperature data.

The environment 2b outgassing rates are generally higher and more persistent than the 2c data, as would be expected. The slope of the log-log plots are in the range of 0 to -0.5. In some instances the 2c data are higher than the 2b data for very early times. It is suggested that this is because the absorbed gas measured in 2c is removed very early in the course of the 2b tests.

The outgas measured in 2b originates mainly from sources which do not release gas at ambient temperature.

The 4a and 4b data are anomalous in that they indicate outgassing rates of the same order as the 2c rates, whereas it would be expected that the measured rates would be between one and two orders of magnitude lower.

This effect is probably due to the use of test chambers at one temperature with the pumping system at a different temperature for the following reason.

The outgassing data were calculated using equation 4-5, which assumes the only way the pressure can drop is for the gas molecules to leave the test chamber. This assumption is true when the entire system is isothermal, i.e., the ambient temperature environment 2c. However, for the high temperature tests, i.e., environment 2b at 200°F (93°C), gas evolved from the sample and the warmer part of the chamber can be condensed on the cooler parts of the system or can leave the chamber via the evacuation line. This condensation would result in measured outgassing rates lower than the true figure by an unknown amount equal to the condensation rate.

The same process of condensation can occur during the preconditioning phase of environments 4a and 4b. Here, however, the effect is more insidious. It is possible that a substantial fraction of the gas evolved from the sample during the 6 hour hot prepumping at 200°F (93°C) is not removed from the chamber, but is instead condensed on cooler portions of the system. Then, when the outgassing test is performed with the chamber and sample at the same temperature, the outgassing rate measured is that of the sample plus the evaporation of the previously condensed gas from the system walls. This measured rate may be very much higher than that of the preconditioned sample, and, if the magnitude of the condensation effect is substantial, could even approach the rate from the unpreconditioned sample. Inspection of 4a and 4b data suggest that this process does seem to be occurring, inasmuch as the 4a and 4b data seem to be generally similar to the 2c data. The accuracy of the 4a and 4b data must therefore be seriously questioned.

From the above discussion, it is apparent that an outgassing apparatus which could be made isothermal over a wide range of sample temperatures is desirable, but such an apparatus would be very costly and has not yet been developed. It is concluded, therefore, that the somewhat disappointing nature of the tests performed under environments 4a and 4b was probably due to them being too demanding for the general state of outgassing test technology.

## 6.6 RECOMMENDED MATERIALS FOR USE IN THE TEST ENVIRONMENTS

The prior sections (6.1 through 6.4) have discussed significant test changes that have occurred in material properties following exposure to the test environments. This section will discuss those materials where no or small property changes have occurred and provide recommendations on materials suitable for use in the different environments.

The recommendations are broken down into a three-step process. First, if oxygen or fluorine propellants or reactants are used, those materials chemically compatible with these propellants are selected. Secondly, the intended use of the insulation system is examined and those materials are selected which show minimal property changes for that usage (i.e., vacuum system, purged system or unprotected insulation system). Finally, the specific environments to which the insulation materials will be subjected are examined and materials showing minimal property changes are selected.

For the candidate materials selected from this screening process, absolute property values from Volume II can then be used to (1) make final material selections, (2) set system requirements, and (3) as input for calculating system performance.

### 6.6.1 Suitable Materials For Use With Fluorine or Oxygen

For those applications where fluorine or oxygen is used in liquid or gas

form, there is always the possibility of gas leakage from the tank, plumbing lines or components into the insulation system. Hazards resulting from chemical combustion of the insulation materials with these oxidizers must be avoided. Table 19 provides a list of those materials that do not ignite under the conditions and exposure times listed.

For fluorine environments, the double gold coated shields are the safest materials to use followed by the double coated aluminized shields and finally the single coated Kapton shield. This order is based on property changes noted later for the D-A-M and S-A-K films in a moist air/fluorine environment (8h) and the fact S-G-K film did ignite even if the S-A-K did not.

For the spacers, SN does not ignite in a dry fluorine environment but does ignite in a moist air/fluorine atmosphere. The sizing on the SN which provides the dry fluorine protection is partially dissolved by the moist conditions exposing the SN to attack.

#### 6.6.2 Suitable Materials for Use in Vacuum, Purged or Unprotected Insulation Systems

After selecting materials compatible with the intended propellant usage, these material selections are narrowed further based on the insulation system usage as shown in Table 20 and described below.

The vacuum jacketed systems shown in the table see pressures below  $10^{-5}$  torr ( $1.33 \times 10^{-3}$  n/m<sup>2</sup>) and temperatures from 660°R (366°K) to 37°R (21°K).

The expendable purge systems experience the vacuum environment described above plus inert gas purges in operation, high humidity and possible water condensation exposures prior to the flight.

The reusable purged system experiences 200°F (93°C) temperatures at ambient pressure during the reentry phase in addition to the previously listed purge environments.

Table 19

INSULATION MATERIALS THAT WILL NOT IGNITE WITH GASEOUS OXYGEN AND FLUORINE UNDER LOW FLOW RATE CONDITIONS\*

Insulation Materials	Oxygen at 70°F (21°C)			Fluorine		
	Without Ignition Source		With Ignition Source**	70°F (21°C)	70°F (21°C)	95% R.H. Air/F <sub>2</sub> at 95°F (35°C) for 4 Hours
	≤ 10 <sup>-3</sup> torr** (.133n/m <sup>2</sup> ) ≤ 150 Days	≤ 760 torr (10 <sup>5</sup> n/m <sup>2</sup> ) ≤ 30 Min		≤ 10 <sup>-3</sup> torr (.133n/m <sup>2</sup> ) ≤ 150 Days	≤ 760 torr (10 <sup>5</sup> n/m <sup>2</sup> ) ≤ 30 Min	≤ 10 <sup>-3</sup> torr F <sub>2</sub> (.133n/m <sup>2</sup> ) 0-760 torr F <sub>2</sub> (10 <sup>5</sup> n/m <sup>2</sup> )
<u>Radiation Shields</u>						
°Single Aluminized Mylar	✓	✓	✓	✓	✓	✓
°Double Aluminized Mylar	✓	✓	✓	✓	✓	✓
°Single Aluminized Kapton	✓	✓	✓	✓	✓	✓
°Single Goldized Mylar	✓	✓	✓	✓	✓	✓
°Double Goldized Mylar	✓	✓	✓	✓	✓	✓
°Single Goldized Kapton	✓	✓	✓	✓	✓	✓
<u>Spacers</u>						
°Silk Net (with sizing)	✓	✓	✓	✓	✓	✓
°Nylon Net	✓	✓	✓	✓	✓	✓
°Dacron Net	✓	✓	✓	✓	✓	✓
°Tissuglas	✓	✓	✓	✓	✓	✓
<u>Ground-Hold Insulation Mtls</u>						
°Beta Glass Cloth	✓	✓	✓	✓	✓	✓
°Narmco 1343/7139 Adhesive	✓	✓	✓	✓	✓	✓
°Goodyear Pliobond 4001/4004 Adh	✓	✓	✓	✓	✓	✓
°Polyurethane Foam	✓	✓	✓	✓	✓	✓
<u>Miscellaneous Insulation Mtls</u>						
°Polyester Velcro Fasteners	✓	✓	✓	✓	✓	✓
°TFE Teflon Film	✓	✓	✓	✓	✓	✓
°Thermatrol Paint	✓	✓	✓	✓	✓	✓
°on Aluminum	✓	✓	✓	✓	✓	✓
°on Fiberglass	✓	✓	✓	✓	✓	✓
°Series 400 Black Paint	✓	✓	✓	✓	✓	✓
°on Aluminum	✓	✓	✓	✓	✓	✓
°on Fiberglass	✓	✓	✓	✓	✓	✓

\* Possible insulation/GO<sub>2</sub>/GF<sub>2</sub> contact is based on occurrence of tank or plumbing leakage. Survival of material (not property changes) is the basis for the recommendations shown.\*\* Recommendations are based on the equivalent fluorine test results. These conclusions may be too conservative since F<sub>2</sub> is a stronger oxidizer than O<sub>2</sub>.

✓ Recommended for use in the environments shown. (Absence of a ✓ indicates the material is not recommended for use in the environments shown.)

The expendable, unprotected insulation system is exposed to the vacuum, high humidity and water condensation environments listed previously in addition to a salt air environment.

The reusable, unprotected insulation system is exposed to those environments listed for the expendable system plus a 200°F (93°C) temperature at ambient pressure during the reentry phase.

Using these definitions, the materials recommended for use in these different environmental combinations is given in Table 20.

#### 6.6.3 Suitable Materials for Use in Specific Environments for Different Exposure Times

For those materials that were selected for a particular insulation system application from the previous section, the specific environments and exposure times of interest are examined next in Table 21. The longest exposure usage time recommended for each material is noted in the table. For those materials that experienced some small property changes, these changes are provided in the table so a judgment can be made on whether the change is acceptable for the application of interest. Finally, for the candidate insulation materials surviving the above listed three step screening process, the absolute property test values provided in Volume II are used (1) to make the final material selections, (2) to set system requirements, and (3) as inputs to calculate both the thermal and mechanical performance of the system.

#### 6.6.4 Sample Calculation (Effect of Exposure on Heat Flux)

The following example is provided to demonstrate how the property changes measured in this program can be used as input to calculate changes in insulation heat flux.

From Ref 7, the heat flux equation for double goldized Mylar with two layers



Table 20

**RECOMMENDED INSULATION MATERIALS FOR USE IN DIFFERENT INSULATION SYSTEMS  
(BASED ON MINIMAL PROPERTY CHANGES)**

Applicable Exposure Environment(s) Insulation Materials	Vacuum Jacketed Insulation System 2	Purged Insulation Systems		Unprotected Insulation System	
		Expendable 2, 4, 5, 7	Reusable 2,3,4,5,7	Expendable 2, 5, 6, 7	Reusable 2,3,5,6,7
<b>Radiation Shields</b>					
°Single Aluminized Mylar	— ✓	✓ <sup>(1)</sup>	✓ <sup>(1)</sup>		
°Double Aluminized Mylar	— ✓	✓ <sup>(1)</sup>	✓ <sup>(1)</sup>		
°Single Aluminized Kapton	— ✓	✓	✓		
°Single Goldized Mylar	— ✓	✓	✓	✓ <sup>(3)</sup>	✓ <sup>(3)</sup>
°Double Goldized Mylar	— ✓	✓	✓	✓	✓
°Single Goldized Kapton	— ✓	✓	✓	✓	✓
<b>Spacers</b>					
°Silk Net (with sizing)	— ✓	✓ <sup>(2)</sup>	✓ <sup>(2)</sup>	✓ <sup>(2)</sup>	✓ <sup>(2)</sup>
°Nylon Net	— ✓	✓	✓	✓	✓
°Dacron Net	— ✓	✓	✓	✓	✓
°Tissuglas	— ✓				
<b>Ground Hold Insulation Materials</b>					
°Beta Glass Cloth	— ✓	✓	✓	✓	✓
°Narmco 7343/7139 Adhesive	— ✓	✓	✓	✓	✓
°Goodyear Pliobond 4001/4004 Adh	— ✓				
°Polyurethane Foam	— ✓	✓		✓	
<b>Miscellaneous Insulation Mtls</b>					
°Polyester Velcro Fasteners	— ✓	✓	✓	✓	✓
°TFE Teflon Film	— ✓	✓	✓	✓	✓
°Thermatrol Paint(External)					
°on Aluminum	— ✓	✓	✓		
°on Fiberglass	— ✓	✓	✓		
°Series 400 Black Paint (External)					
°on Aluminum	— ✓	✓	✓	✓	✓
°on Fiberglass	— ✓	✓	✓	✓	✓

- (1) Although the humidity (Environment 5) and water immersion (Environment 7) exposures caused no significant changes in adhesion or emittance properties for these radiation shields, Refs 1 and 6 indicate repeated water condensation will remove the aluminum coating. Therefore, before these materials are selected for a particular application, a tradeoff should be made between the costs of using the more expensive gold coated shields vs the added cost of maintaining a dry, gas environment in the aluminized insulation.
- (2) The layer density of nylon net increases 16% after water immersion; otherwise, minimal property changes occur.
- (3) The gold adhesion is affected by the salt air environment (-26%); otherwise, minimal property changes occur.
- ✓ Recommended for use. (Absence of a ✓ indicates the material is not recommended for use in the systems shown.)

Table 21

## MATERIALS RECOMMENDED FOR USE IN THE ENVIRONMENTS BASED ON MINIMAL PROPERTY CHANGES

MATERIAL ENVIRONMENT	EXPOSURE PARAMETERS		RADIATION SHIELDS				SPACERS			GROUND-HOLD MTL'S				MISCELLANEOUS MTL'S			
	O <sub>2</sub> OR F <sub>2</sub> P.P. TORR	TEMP °R (°K) HR.	S-A-M	D-A-M	S-A-K	S-G-M	D-G-M	S-G-K	SILK NYLON NET	TESSU- GLAS NET	BETA GLASS CLOTH	ADHESIVES 7343/4001/ FOAM 2 PCF	VELCRO	TFE TEFLON	ON F.G.	ON F.G.	ON F.G.
SIMULATED ATMOSPHERIC ENVIRONMENTS																	
HIGH TEMPERATURE 200° F (93° C) 40% R.H.	3a	24															
	3b	72															
	3c	240															
	5a	12															
95% R.H. AT 95° F (35° C)	5b	24															
	5c	72															
	6a	12															
	6b	24															
95% R.H./SALT AIR AT 95° F (35° C)	6c	72															
	7a	0.5															
	7b	2															
	7c	24															
WATER IMMERSION AT 70° F (21° C)	8g	10 <sup>-3</sup>															
	8h	760															
SIMULATED ORBITAL ENVIRONMENTS																	
GASEOUS FLUORINE AT 70° F (21° C)	8c	10 <sup>-3</sup>															
	8f	100															
		3600															
GASEOUS OXYGEN AT 70° F (21° C)	8c	10 <sup>-3</sup>															
	8d	100															
		3600															
VACUUM ≤ 10 <sup>-6</sup> TORR	2a	660 (366)															
	2b	660 (366)															
	2c	530 (295)															
	2d	140 (78)															
2c		37 (21)															

NOTES:

1) ✓ RECOMMENDED FOR USE IN THE ENVIRONMENT SHOWN

2) BASED ON MINIMAL PROPERTY CHANGES.

3) PROPERTY SYMBOL ( ) RECOMMENDED FOR USE IF PERCENT CHANGE IN THE

PROPERTY VALUE SHOWN IS SATISFACTORY FOR INTENDED USE.

4) NT - NOT TESTED

5) LONGEST RECOMMENDED EXPOSURE TIME IS SHOWN FOR EACH MATERIAL.

WHERE

α SOLAR ABSORBANCE

AD COATING ADHESION

F<sub>c</sub> COMPRESSIVE STRENGTHF<sub>u</sub> ULTIMATE SHEAR STRENGTHF<sub>t</sub> ULTIMATE TENSILE STRENGTH

LD LAYER DENSITY

W WEIGHT

of silk net spacers between each shield is given by:

$$q = \frac{4.37 \times 10^{-11} (\bar{N})^{3.27} T_m}{N_s + 1} (T_H - T_C) + \frac{3.12 \times 10^{-11} \epsilon_{TR}}{N_s} T_H^{4.51} - T_C^{4.51} \quad (6-1)$$

where

- $q$  = total heat flux through the insulation, Btu/hr ft<sup>2</sup> (w/m<sup>2</sup>)
- $\bar{N}$  = layer density of the insulation system, no. of radiation shields/in. (no. of radiation shields/cm)
- $N_s$  = number of radiation shields in an insulation system, dimensionless
- $T_m$  = absolute mean temperature between  $T_C$  and  $T_H$ , °R (°K)
- $T_C$  = absolute temperature of the cold boundary, °R, (°K)
- $T_H$  = absolute temperature of the hot boundary, °R (°K)
- $\epsilon_{TR}$  = total hemispherical emittance of an insulation radiation shield surface, dimensionless

Assume a 100 layer ( $N_s$ ) D-A-M/silk net insulation system has been exposed to a preconditioning 200°F (93°C) high vacuum environment for 240 hours (Envir. 2b) and is then flown in orbit where  $T_H = 400^\circ\text{R}$  (222°K) and  $T_C = 37^\circ\text{R}$  (21°K). Also, assume the initial layer density ( $\bar{N}$ ) is 60 radiation shields/in. (24 radiation shields/cm).

To calculate the percentage decrease (improvement) in  $q$  due to the bakeout exposure, the following formula is used:

$$\text{Percent change} = \frac{q_{\text{post}} - q_{\text{pre}}}{q_{\text{pre}}} (100) \quad (6-2)$$

where  $q_{\text{pre}}$  refers to the heat flux value that would be obtained prior to the bakeout exposure and  $q_{\text{post}}$  refers to the heat flux value obtained after the exposure. From Volume II, the mean control value of  $\epsilon_{\text{pre}}$  for 16 D-G-M specimens is .024. After 240 hours of high temperature vacuum exposure, the  $\epsilon$  value has decreased a maximum of -40% (from Table 12) apparently due to a

cleaning action on the gold surface so the post exposure  $\epsilon_{\text{post}}$  value is

$$\epsilon_{\text{post}} = \epsilon_{\text{pre}} - .4\epsilon_{\text{pre}} = .014$$

Using the appropriate  $\epsilon$  values in equation 6-1 and calculating the percent decrease in heat flux rate from equation 6-2 gives a -6.5 percent total decrease in  $q$  for a 40 percent decrease in gold emittance.

The change in layer density of silk net ( $\bar{N}$ ) due to the environmental exposure could also be factored in to these calculations but was excluded for the following reason. The test results showed  $\bar{N}$  decreased 31 percent following the exposure, that is the layers evidently became more rigid and the total free standing height of the silk net increased 31 percent. This decrease in layer density would also decrease  $q$ . However, in an actual insulation installation, the layers are constrained at a specified thickness by buttons, joint closures etc so the layer density can increase (be compressed) but can not decrease (expand) very much due to the physical attachment methods used.

## Section 7

### CONCLUSIONS

In this program, twenty different insulation or insulation related materials were exposed to eight different conditions (including a control environment for reference) representing operational environments that include different combinations of high humidity, salt air, water immersion, various operational temperatures, space vacuum, space-vented propellants and tank leakage. The effect of these environments on the specified properties are determined as a function of exposure time, exposure temperature or in some cases fluorine or oxygen partial pressure. The major conclusions that were arrived at, based on the test results of the program, are discussed in the four material groupings of radiation shields (6 each), spacers (4 each), ground-hold materials (4 each) and miscellaneous materials (6 each).

7.1 RADIATION SHIELDS [Single aluminized mylar (S-A-M), double aluminized mylar (D-A-M), single aluminized kapton (S-A-K), single goldized mylar (S-G-M), double goldized mylar (D-G-M), and single goldized kapton (S-G-K)]

Significant conclusions based on the test results are provided for the above listed aluminized and goldized films on mylar and kapton substrates.

- ° All the shields are recommended for use in vacuum systems.
- ° The gold coated shields are recommended for use in purged and unprotected systems.
- ° D-A-M and S-A-K are suitable for use in purged systems if dry environments are maintained prior to flight.
- ° D-G-M and D-A-M are recommended for use in insulation systems which may be accidentally exposed to dry fluorine environments

up to one atmosphere in pressure. (Double coated Kapton films were not tested but are also probably satisfactory for use in this environment.)

- ° D-G-M is recommended for use in insulation systems which may be accidentally exposed to moist air/fluorine environments at one atmosphere partial pressure. (Double goldized Kapton was not tested but is also probably satisfactory for use in this environment.)
- ° All the shields are suitable for use in insulation systems which may be exposed to less than  $10^{-3}$  torr ( $.133 \text{ n/m}^2$ ) fluorine or oxygen pressure for up to 150 days.
- ° Gold coated films are recommended for use in salt air and high humidity environments.
- ° D-A-M and S-A-K aluminum coated films can be used in high humidity environments for up to 240 hours; however, testing of the films in the expected environment is recommended before this choice is made.
- ° Emittance values of gold coated substrates can be lowered (improved) 29 to 52% by a 240 hour 200°F (93°C) vacuum exposure.
- ° Kapton outgasses less than Mylar.
- ° Gold outgasses less than aluminum.
- ° Double coated Mylar outgasses less at 200°F (93°C) than single coated Mylar.

- ° The outgassing rate of all shields is essentially zero at 140°R (77°K).

7.2 SPACERS [ Silk net (SN), Nylon net (NN), Dacron net (DN), Tissuglas (TG)]

Significant conclusions based on the test results are provided for the above listed low conductivity spacers.

- ° All four spacers are recommended for use in vacuum systems.
- ° DN and NN are recommended for use in purged or non-purged insulation systems.
- ° DN exhibited the fewest property changes of the four spacers tested.
- ° SN is recommended for use in insulation systems which may be accidentally exposed for short periods of time to dry fluorine environments up to one atmosphere in pressure; none of the spacers are recommended for use in prolonged (150 days) fluorine environments at  $10^{-3}$  torr ( $.133 \text{ n/m}^2$ ) pressure.
- ° None of the spacers are recommended for use in a moist air/fluorine environment at one atmosphere partial pressure.
- ° DN outgasses less than the other spacers over the temperature range 70°F (21°C) to 200°F (93°C) on an overall integrated basis.
- ° SN outgasses less than the other spacers when preconditioned at 200°F (93°C).



- ° The outgassing rate of all spacers is essentially zero at 140°R (77°K).

7.3 GROUND-HOLD MATERIALS [Beta glass cloth (B), Narmco 7343/7139 adhesive (NA), Goodyear 4001/4004 adhesive (GY), polyurethane foam (PF)]

Significant conclusions based on the test results are provided for the above listed glass cloth, adhesives and foam.

- ° B and NA are recommended for use in vacuum, purged and non-purged insulation systems.
- ° PF is recommended for use in expendable purged and expendable non-purged insulation systems.
- ° GY adhesive is recommended for use in insulation systems which may be accidentally exposed to short term dry fluorine environments up to one atmosphere in pressure.
- ° None of the materials are recommended for use in a moist air/fluorine environment at one atmosphere partial pressure or in a  $10^{-3}$  torr ( $.133 \text{ n/m}^2$ ) fluorine environment for 150 days.
- ° The outgassing rate of all the ground-hold materials is essentially zero at 140°R (77°K).

7.4 MISCELLANEOUS MATERIALS [Velcro fasteners (V), Teflon film (TFE), Thermatrol paint on aluminum (TH-A), Thermatrol paint on fiberglass (TH-F), 3M black paint on aluminum (BL-A), 3M black paint on fiberglass (BL-F)]

Significant conclusions based on the test results are provided for the above listed fastener, film and thermal control paints.

- ° All the materials are recommended for use in vacuum and purged insulation systems.
- ° V, TFE, BL-A and BL-F are recommended for use in unprotected insulation systems.
- ° TFE is recommended for use in any of the low flow rate fluorine environments, dry or wet.
- ° BL-A and BL-F properties are extremely stable in all the environments (except fluorine exposure at one atmosphere).
- ° The outgassing rate of all the miscellaneous materials is essentially zero at 140°R (77°K).

## Section 8

### REFERENCES

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